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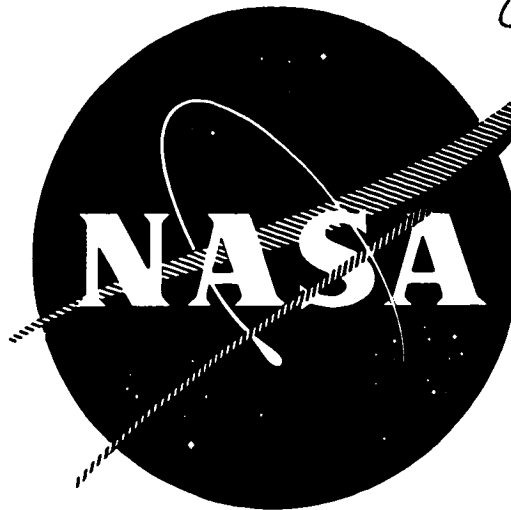
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**RESEARCH AND DEVELOPMENT PROGRAM ON MAGNETIC,  
ELECTRICAL CONDUCTOR, ELECTRICAL INSULATION,  
AND BORE SEAL MATERIALS**

Second Quarterly Report

by

P. E. Kueser et al

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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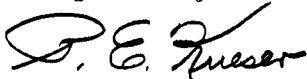
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## PREFACE

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## ABSTRACT

This is the second quarterly report concerning the evaluation of design information on materials at elevated temperature for application to advanced space electric power systems. A "test book" identifying the test program, the specimen geometry, the test atmospheres, and the testing procedures has been prepared.

Mechanical tests were initiated on magnetic materials. Cube-textured materials have shown a variation in Poisson's ratio depending upon the orientation of the measurement in respect to the crystal lattice. Hiperco 27 was tested at elevated temperatures in both argon and air atmospheres and no significant difference in tensile or creep properties was observed.

A number of organic and inorganic insulation materials are presented. These include most configurations suitable for electric apparatus.

A new conductor, beryllia dispersion-strengthened copper, has been fabricated. It exhibits stress-rupture properties a factor of four better than beryllium-copper No. 10 at elevated temperature. Beryllium-copper is presently one of the better high-strength, high-conductivity materials available.

The bore seal effort has been focused on the development of metalized ceramic-to-metal sealing systems prior to their planned alkali-metal exposure tests. During the next quarter, the test program will be accelerated.

*Author*

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## SECTION I

### INTRODUCTION

This is the second quarterly report concerning magnetic, electrical insulating, electrical conductor and bore seal materials needed for designing advanced space electric power systems. The first report identified over 700 references which were considered pertinent to this area and indicated the gaps which occur in the necessary design information. Guidelines were also prepared which outlined the material properties of interest to designers of this equipment.

The second quarter of effort has been primarily spent in defining the test specifications, specimen drawings, in ordering materials, in checking out equipment and in preparing the liquid-metal loading and handling facilities. A test book outlining each test, the appropriate test specification, the specimen drawings, and the test environment has been prepared. It is being used as the master document for supporting the test program.

All magnetic materials have been received except Maraging steel. The Maraging steels required screening tests to determine the nickel content necessary to give the desired combination of magnetic and mechanical properties. Mechanical tests have been initiated on the magnetic materials.

Several of the conductor materials have been delivered. One of these, dispersion-strengthened copper (BeO), represents a newly developed material. The remaining conductors, which are austenitic stainless or inconel clad, include dispersion hardened copper, zirconium copper, and silver cores. These are under development for this program and will be delivered by the middle of April.

Approximately ninety percent of the insulation materials are available or will be available by April 1.

Selection of specific types has taken considerable judgement as there are a number of similar insulating materials available from which the most representative sample must be chosen. The uncertain availability of pyromellitic dianhydride (PMDA) has also held up the final selection of insulation materials. In the future, this compound will not be available to industry and all epoxy materials using it have been eliminated from the program.

The bore seal effort has been directed towards the development of ceramic-to-metal sealing systems prior to their alkali metal exposure tests.

A technical discussion concerning these areas is given in the following sections.

## SECTION II

### MAGNETIC MATERIALS

#### A. INTRODUCTION.

Selection of the magnetic materials for the present program has been based on applications to both solid and laminated rotors where strength is important; to motor and generator stators, electromagnetic pumps and transformers where high induction and low losses at a reasonable magnetizing force are desired; to d-c solenoids; and to saturable reactors where "square-loop" magnetic properties are desirable. The materials have been further selected for their elevated temperature capabilities and their potential application to advanced space electric power systems.

#### B. SUMMARY OF EFFORT IN THIS QUARTER.

Emphasis has been placed in procuring the magnetic materials, developing rolling and heat treating methods, machining and punching specimens, application of interlaminar insulations where required, and the winding of test samples. Mechanical testing has also been initiated and the theoretical analysis of magnetic properties continued.

## C. DISCUSSION OF MAGNETIC PROPERTIES OF CUBE-ORIENTED MATERIALS.

Some basic and high temperature d-c magnetic properties of Cubex magnetic alloy will be discussed\*. The Cubex alloy is a 3% silicon-iron material with superior magnetic properties parallel and perpendicular to the rolling direction; this makes it especially useful in applications where reductions in size and weight are at a premium.

### 1. Basic Considerations

Most magnetic materials are magnetically anisotropic, that is, the magnetic properties are different along different crystallographic directions of the crystal lattice. In iron, and most of its magnetic alloys, the edges of the cubic lattice, usually referred to as the  $\langle 001 \rangle$  directions, are the directions of highest permeability and lowest losses, the easiest directions to magnetize. By proper metallurgical treatments, magnetic sheets and tapes with a proper texture in their grains were developed to take advantage of this anisotropy<sup>1, 2, 3</sup>.

In the 1930's a texture, referred to as a "cube-on-edge" orientation, was developed by Goss in approximately 3% silicon-iron sheets. In this type of orientation, the crystals of the sheet are oriented in such a way that the majority have a cube edge parallel to the rolling direction of the sheet and a (110) plane (combining two edges and two face diagonals of the cube) parallel to the plane of the sheet. Thus, one easy direction of magnetization is parallel to the rolling direction. Electrical equipment such as transformers were designed to take advantage of the easy direction of magnetization produced in the sheet. However, there are some limitations inherent in this material. The (110) planes of the crystal lattice

\*Testing and data evaluation were performed by J. J. Clark and N. Pavlik of Westinghouse Research Laboratories. (Westinghouse sponsored program Report No. 63-141-319-R5.)

<sup>1</sup>Assmus, F.; et al, "Cube-Textured Silicon-Iron" (in German), Zeitschrift für Metallkunde, V. 48, p. 341, June 1957.

<sup>2</sup>Wiener, G. W.; Detect, Klaus, "A Major Advance in Magnetic Materials", Journal of Metals, AIME, p. 507, August 1958.

<sup>3</sup>Walter, J. L.; Hibbard, W. R.; Fiedler, H. C.; Grenoble, H. E.; Pry, R. H.; Frischmann, P. G.; "Magnetic Properties of Cube-Textured Transformer Sheet", Journal of Metals, p. 509, August 1958.

are parallel to the plane of the sheet which places a  $\langle 110 \rangle$  direction at  $90^\circ$  and a  $\langle 111 \rangle$  direction at  $55^\circ$  to the rolling direction in the plane of the sheet. Since the  $\langle 110 \rangle$  and  $\langle 111 \rangle$  directions are harder directions of magnetization than the  $\langle 001 \rangle$  direction, higher losses and lower permeability result when the flux travels at an angle to the rolling direction. This occurs, for instance, when the flux flows in a radial direction in the rotor or stator of a motor or generator or at the corners of a lapped-joint transformer core. The increased losses lower the electrical efficiency and increase the amount of heat.

The most practical way to eliminate the high losses in the cross direction would be to arrange the crystal orientation of the material so that two  $\langle 001 \rangle$  directions lie in the plane of the sheet, parallel and perpendicular to the rolling direction ("cube-on-face" orientation). The "cube-on-face" texture was just recently developed for 3% silicon-iron and is considered a major breakthrough in magnetic materials used for power applications. The formation of this texture depends on what is believed to be a new mechanism of crystal growth and on carefully controlled structure during the metallurgical processing.

Figure II-1 gives an illustration of the merits of "cube-on-face" orientation in applications where magnetic flux flows in more than two directions such as in the stator and rotor cores of a motor or a generator. Calculated average magnetization curves are shown for both "cube-on-face" and "cube-on-edge" orientations for a stack of 3% silicon iron punchings cut in various directions. The applied field considered in this calculation is 10 to 250 oersteds. As can be seen, the "cube-on-face" orientation yields, for the same magnetizing force, flux densities approximately 15% higher than those pertaining to the "cube-on-edge" magnetization curve. Furthermore, more uniformity with respect to angular positions can be expected from the "cube-on-face" orientation than from the "cube-on-edge" orientation because the former has two easy  $\langle 001 \rangle$  directions of magnetization but no "hard"  $\langle 111 \rangle$  direction in the plane of the sheet.

#### Samples and Tests

Two types of samples each obtained from Cubex steel sheets, 0.011 inch thick, were tested. Ring samples 2-13/16 inch I.D. x 3 inch O.D. were punched from the sheet. The magnetic properties measured on these samples are an average of the properties for all orientations in the sheet. Hollow square samples, 4-31/32 inch outside by 4 inch inside, were punched in such a manner that the adjacent legs were parallel and perpendicular respectively to the rolling direction of the sheet. Magnetic properties measured on these specimens are an average of the straight and cross grain properties of the material. All punchings were deburred and given one coat of Alkophos insulation (aluminium-ortho-phosphate) before heat treatment. After heat treatment, an additional coat of Alkophos was applied for interlaminar insulation.

The specimens were wound as ring samples with primary and secondary windings. DC tests were made by the ballistic method as described in ASTM Method A341. Normal induction curves were determined to a maximum magnetizing force of 250 oersteds. Residual induction and coercive force were measured from a magnetizing force of 10 oersteds.

## 2. Test Results

The effect of temperature on the d-c magnetic properties of Cubex ring laminations in the range of room temperature to 1100°F is displayed in Figures II-2 and II-3 (d-c magnetization curve), and Tables II-1 (coercive force), II-2 (residual induction), and II-3 (maximum permeability). The tables also give the respective values for hollow, square laminations. Figures II-4 and II-5 are intended to compare the d-c magnetic properties at room temperature and 700°F for both configurations of Cubex samples considered in this report - ring and hollow, square laminations.

As can be observed in the illustrations cited above, there is a decrease with increasing temperatures in the coercive force ( $H_c$ ) and remanent induction ( $B_r$ ), as well as in the flux densities ( $B$ ) in fields of 1 oersted and above. This is a pattern generally shown by soft magnetic materials at increasing temperatures provided there is no phase change of either first or second order in the temperature range considered. E. M. Terry<sup>4</sup> reported this general phenomena in his 1910 study of pure iron. More recently C. W. Chen<sup>5</sup>, of Westinghouse Research Laboratories, confirmed this in a thorough study of the temperature effect on the magnetic properties of "cube-on-edge" 3% silicon-iron.

The results reported herein show that the trend, as well as the percentage decrease with temperature in Cubex d-c magnetic properties, are almost identical with those reported by Chen for "cube-on-edge" silicon-iron. The permeability peaks of Cubex ring samples are relatively constant up to 900°F with an increase at 1100°F. The same behavior is shown by Chen for "cube-on-edge" silicon-iron for a field between 0.1 and 0.2 oersteds. These field values correspond to those at which Cubex permeability peaks occurred.

<sup>4</sup>Terry, E. M., "Effect of Temperature on Magnetic Properties of Electrolytic Iron", Physical Review, V. 30, p. 133, 1910.

<sup>5</sup>Chen, C. W., "Temperature Dependence of Magnetic Properties of Silicon-Iron", Journal of Applied Physics, V. 29, p. 1339, Sept. 1958.



TABLE II-1. Effect of Temperature on Coercive Force of  
Cubex Laminations, 0.011 Inch Thick

Sample	Coercive Force (oersteds) ( $B_{tip} = 10$ oersteds)					
	R. T.	300°F	500°F	700°F	900°F	1100°F
Ring #1 *	0.102			0.079		
Ring #2 *	0.157	0.146	0.131	0.110	0.088	0.061
Square	0.134			0.110		
*Rings #1 and #2 were prepared from two different heats and were processed at the Westinghouse Research Laboratories. A complete series of test was carried out on Ring #2 only.						

Reference: Westinghouse Report No. 63-141-319-R5

TABLE II-2. Effect of Temperature on Residual Induction of  
Cubex Laminations, 0.011 Inch Thick

Sample	Residual Induction (gauss) ( $B_{tip} = 10$ oersteds)					
	R. T.	300°F	500°F	700°F	900°F	1100°F
Ring #1	6100			3900		
Ring #2	6150	5500	4800	3850	3475	2800
Square	8300			5200		

Reference: Westinghouse Report No. 63-141-319-R5

TABLE II-3. Effect of Temperature on Typical DC Permeability Peaks of Cubex Laminations, 0.011 Inch Thick

Sample	Permeability Peaks *					
	R. T.	300°F	500°F	700°F	900°F	1100°F
Ring #1	30,260			25,830		
Ring #2	18,250	18,000	17,570	16,490	18,000	25,740
Square	27,080			22,980		
*Permeability peaks are not necessarily maximum permeability because, for practical reasons, the tests were initiated at the field corresponding to an induction of 2 kilogauss.						

Reference: Westinghouse Report No. 63-141-319-R5

As has been shown in literature, especially by M. Kersten<sup>6</sup> and L. Neel<sup>7</sup>, the coercive force is a function of  $\lambda_s/I_s$  or  $K/I_s$  or both ( $\lambda_s$  denotes saturation magnetostrictions,  $K$  crystal anisotropy energy, and  $I_s$  saturation magnetization). As Chen points out, since the anisotropy energy for silicon-iron alloys is considerably larger (by a factor of 260) than the energy associated with magnetostriction, consideration is given to  $K$  and  $I_s$  only. The variation of  $K$  and  $I_s$  with temperature has not been reported for silicon-iron alloys but in view of the resemblance in magnetic properties between silicon-iron alloys and pure iron, it seems justified to assume that silicon-iron follows the same trend in basic magnetic properties as pure iron. Consequently, since crystal anisotropy drops faster and vanishes at lower temperatures than  $I_s$ , a decrease of the coercive force at elevated temperatures is to be expected.

A similar interpretation was given by Chen for the remanence,  $B_r$ . The energy required for the formation of boundaries of new domains, a process which controls the remanence, is also highly temperature sensitive. R. M. Bozorth<sup>8</sup> suggested the following expression for domain wall energy

$$W = 2 \left( \frac{3}{2} \sigma \lambda_s + K/3 \right)^{1/2} \left( \frac{k\theta}{d} \right)^{1/2}$$

where  $\sigma$  is the stress acting on the material,  $k$  the Boltzmann's constant,  $\theta$  the Curie temperature, and  $d$  the interatomic distance. An increase in temperature brings about a decrease in the domain wall energy because of the resulting decrease in  $K$  and therefore a decrease in remanence.

The magnetization curves of both Cubex ring and hollow square laminations show the same pattern with increasing temperatures as that reported by Chen for "cube-on-edge" silicon-iron. The flux densities in fields of 1 kilogauss and above decrease with increasing temperatures whereby at temperatures above 700°F and in fields above 10 oersteds the margin between the magnetization curves increases as the temperature is increased.

<sup>6</sup> Kersten M., The Basis of a Theory of the Ferromagnetic Hysteresis and the Coercive Force, (book in German), S. Hirzel Publishing Co., Leipzig, 1943.

<sup>7</sup> Neel, L., Principles of a New General Theory of the Coercive Field, (in French), Ann. University, Grenoble, V.22, p. 299, 1956.

<sup>8</sup> Bozorth, R. M., "On Magnetic Remanence", Zeitschrift für Physik, V.124, p. 519, 1948.

A  $B_{10}$ \* value of 15.7 kilogausses was obtained at room temperature in Cubex ring samples. As already mentioned, a ring sample integrates magnetic properties of a cube-oriented material, i.e., all angular directions between the cube-edge and the cube-face diagonal. For a completely cube-oriented sample (cube face parallel with the surface of the sheet) integrated directional properties will correspond to those measured in Cubex alloy strips at approximately  $22\frac{1}{2}^\circ$  angular deviation from the cube-edge (or half way between the cube-edge and the cube-face diagonal). This is in agreement with the data by K. Foster and J. J. Kramer<sup>9</sup> who show that the  $B_{10}$  value of 15.7 kilogausses corresponds to a directional deviation of approximately  $22\frac{1}{2}^\circ$  of a completely cube oriented thin sheet when demagnetization effects in thin sheets are considered.

Foster and Kramer used Cubex alloy strips 0.004 inch thick, cut in the rolling direction on which  $B_{10}$  values were determined as a function of angular deviations of cube grains; they developed in good agreement with the experimental data the following expression for  $B_{10}$

$$B_{10} = \frac{B_s}{\cos \theta + \sin \theta}$$

where  $\theta$  is the angle between the nearest cube edge direction and the direction of the applied field. This expression differs from the calculations shown in Figure II-1 in that Figure II-1 refers to the magnetic properties of a single crystal having no demagnetizing effects in which case  $B_{10}$  can be expressed as  $B_{10} = B_s \cos \theta$ .

The hollow square sample of Cubex whose d-c magnetic properties are shown in Figures II-3 and II-4 show a  $B_{10}$  value of 17 kilogausses. It should be remembered that a hollow square in addition to four cube edge directions in its legs also incorporates considerable angular deviations

\* The induction value for a field of 10 oersteds,  $B_{10}$ , is a convenient practical measure of the orientation effect in silicon-iron alloys. The  $B_{10}$  value can also be taken as a good approximation of the magnetization value reached in a sheet when the initial stages of magnetization characterized by domain wall movement have taken place but practically no domain rotation has occurred<sup>9</sup>.

<sup>9</sup>Foster, K.; Kramer, J. J., "Effect of Directional Orientation on the Magnetic Properties of Cube-Oriented Magnetic Sheets", Journal of Applied Physics, V.31, p. 2335, 1960.

in its corners. The total angular deviation of this sample from the cube edge, judging from the Foster-Kramer equation, amounts to between  $10^\circ$  and  $15^\circ$ .

In summary, d-c magnetic properties of cube-oriented 3% silicon-iron follow a pattern generally shown by soft magnetic materials at increasing temperatures, i.e. there is a decrease with increasing temperatures in the coercive force and remanence as well as in the flux density above low field levels. Cube-textured ring laminations displayed a  $B_{10}$  value of 15.7 regardless of angular distribution of cube directions. This feature makes it attractive for applications such as dynamic apparatus where the construction geometry necessitates a changing magnetic path.

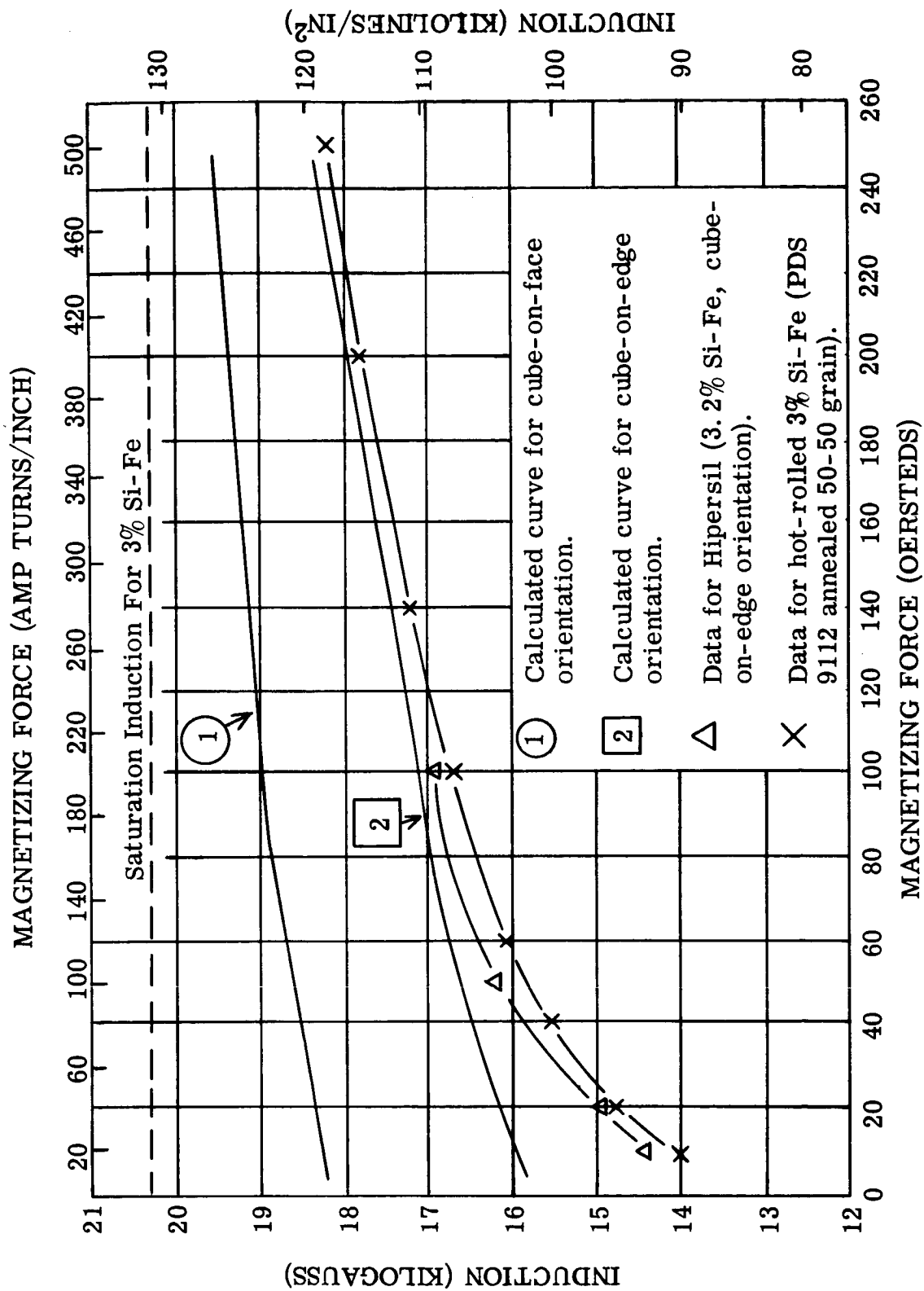


FIGURE II-1. Average Magnetization Curves for Punchings Cut in Random Directions  
From 3% Silicon-Iron Sheet (Taken From the Westinghouse Research  
Memo 60-8-06-28-M-1, R. W. Cole, 10/21/54)

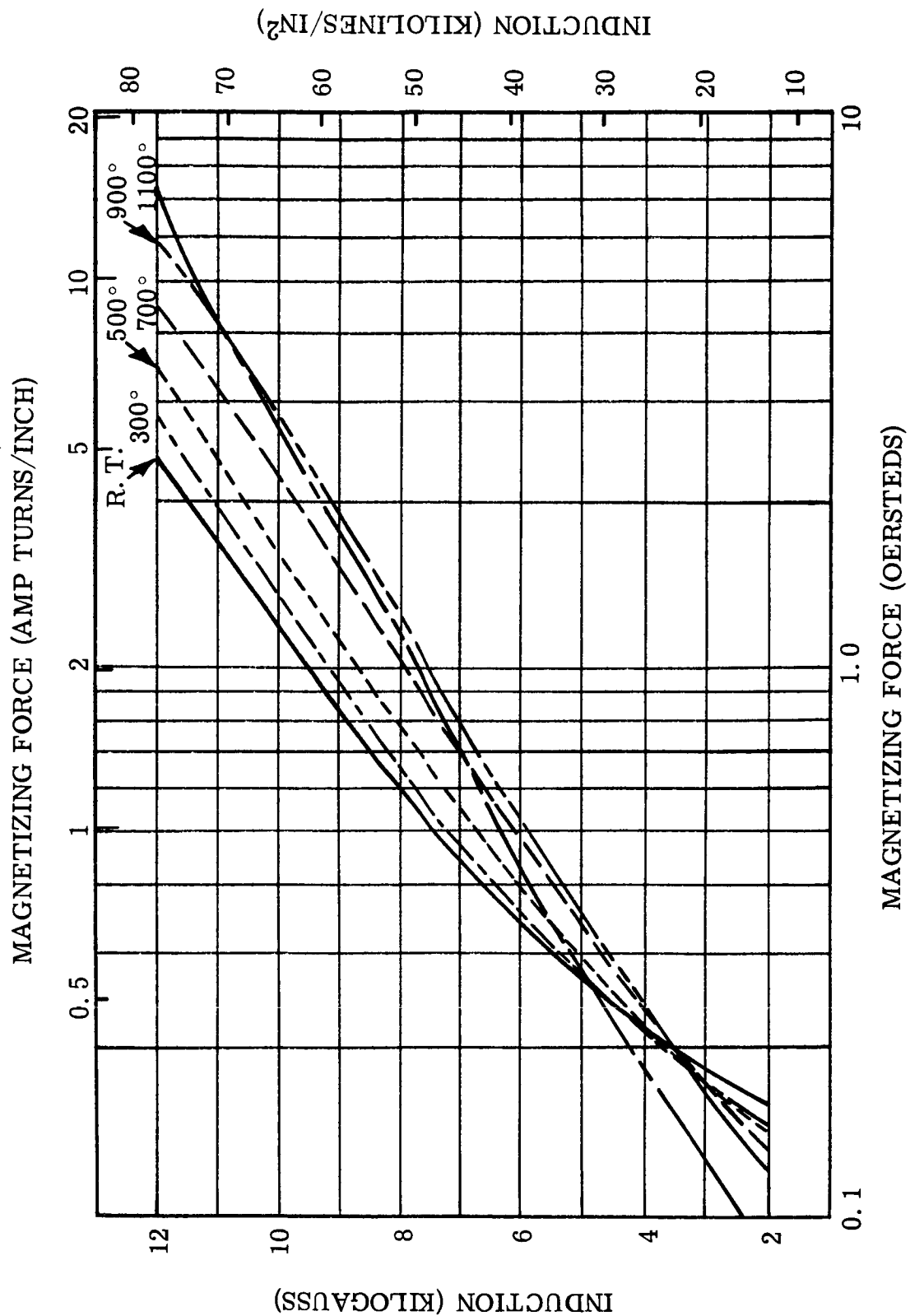


FIGURE II-2. DC Magnetization Curves (Low Inductions) from Room Temperature to 1100°F Cubex Ring (Sample #2), 0.011 inch Lamination Thickness



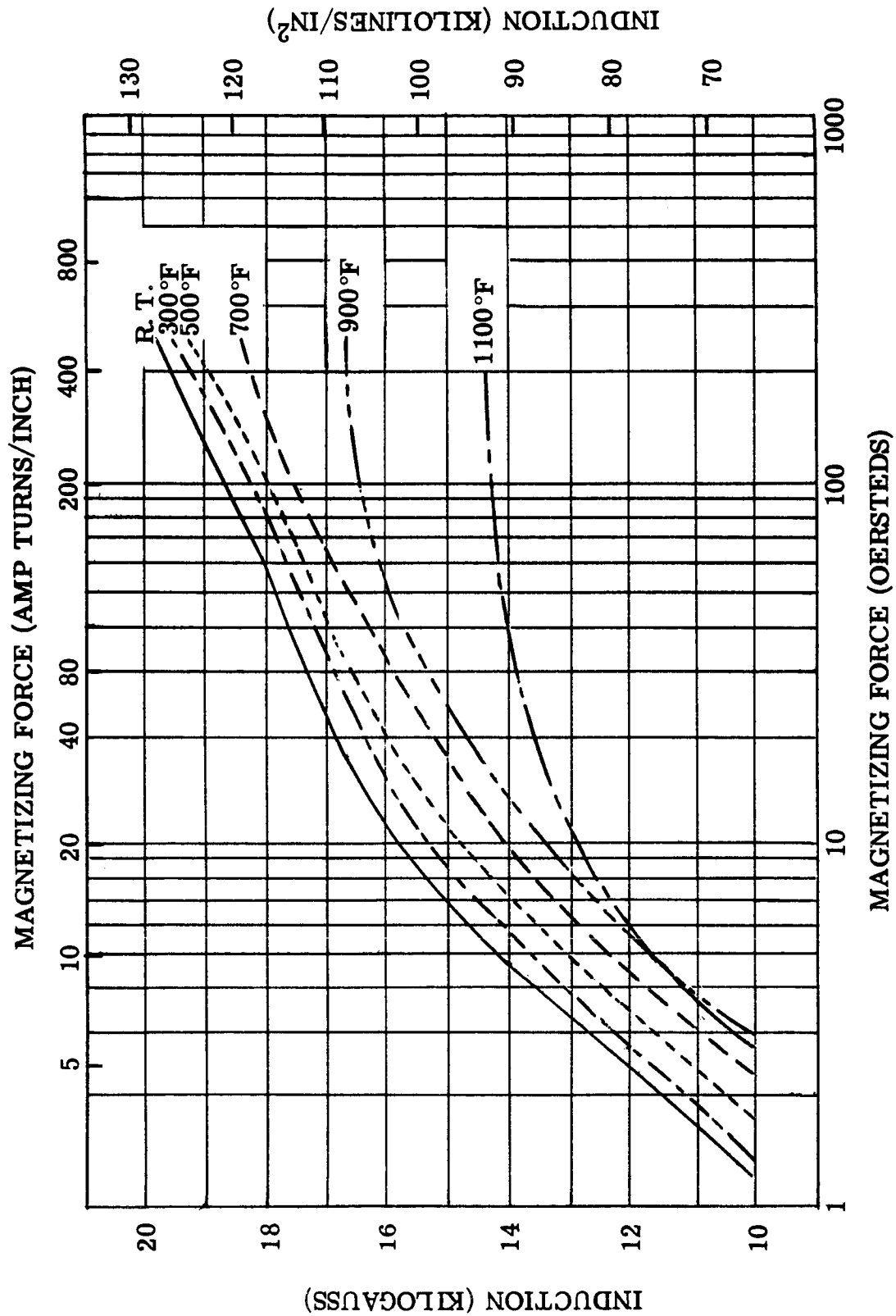


FIGURE II-3. DC Magnetization Curves (High Induction) From Room Temperature to 1100°F Cubex Ring (Sample #2), 0.011 Inch Lamination Thickness (Reference: Westinghouse Report No. 63-141-319-R5)

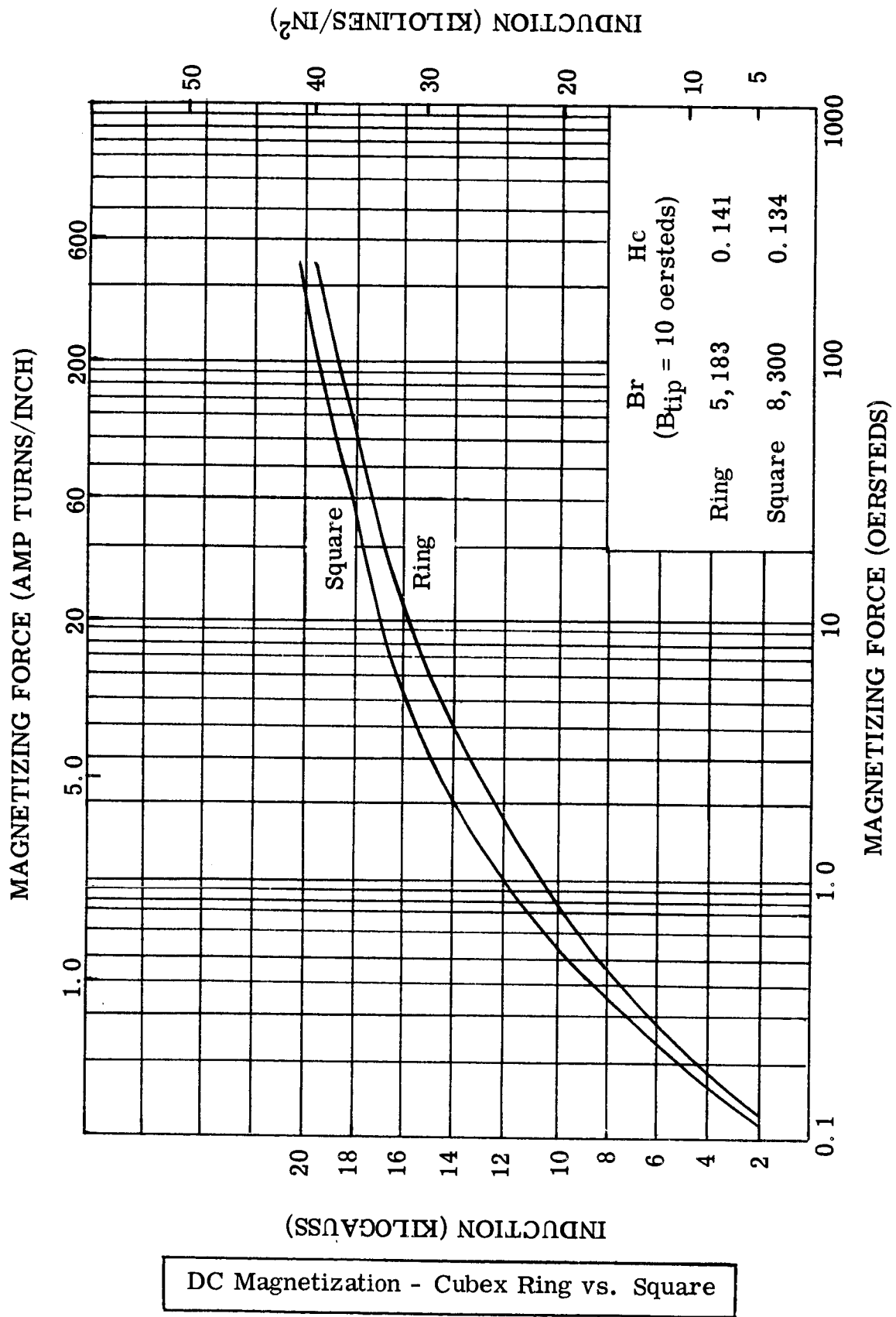
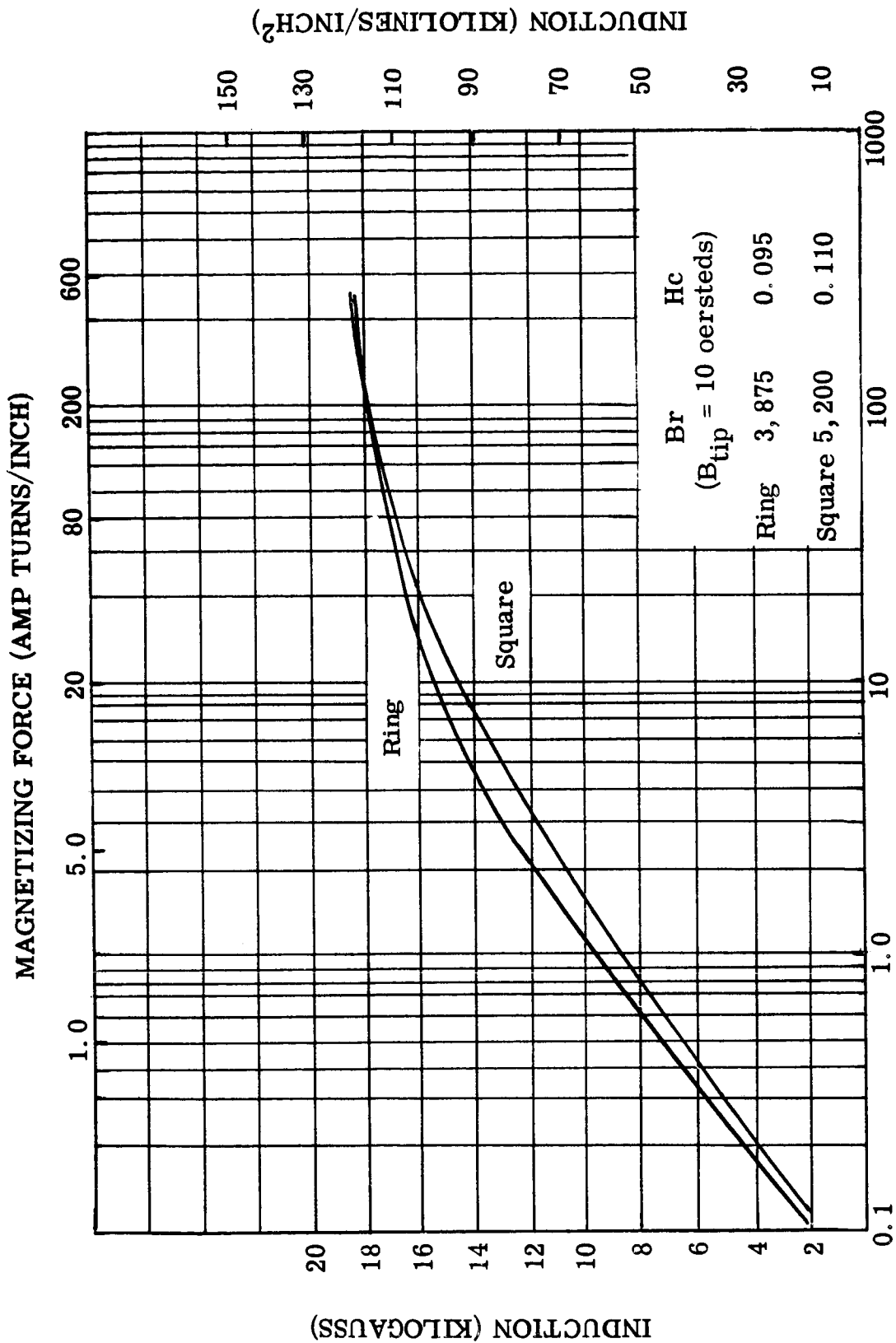


FIGURE II-4. DC Magnetization Curves at Room Temperature, Ring Versus Square  
Cubex, 0.011 Inch Lamination Thickness (Reference:  
Westinghouse Report No. 63-141-319-R5)



DC Magnetization - Cubex Ring vs. Square

MAGNETIZING FORCE (OERSTEDS)

FIGURE II-5. DC Magnetization Curves at 700°F, Ring Versus Square Cubex, 0.011 Inch Lamination Thickness (Reference: Westinghouse Report No. 63-141-319-R5)

D. MATERIAL PROCUREMENT AND PREPARATION.

1. Material Procurement

A list of the magnetic materials, their sources, and purchasing specifications is provided below.

<u>Material and Form</u>	<u>Source</u>	<u>Product Specification</u>
(W) Cubex alloy sheet and tape wound cores. (3Si - Fe)	Westinghouse Research and Development Center, Pittsburgh 35, Pa., and Westinghouse Specialty Transformer Division, Beaver, Pa.	No commercial specification available.
Supermendur tape, sheet and 2V-Permendur bar. (49Fe - 49Co - 2V)	Arnold Engineering Corp. Marengo, Ill.	Arnold Engineering Product Specification for Supermendur with Westinghouse core loss requirement.
Hiperco 50 sheet (49Fe - 49Co - 2V)	Westinghouse Materials Manufacturing Dept., Blairsville, Pa.	No commercial specification available.
Hiperco 27 sheet, bar and casting. (27Co - Fe)	As above.	No commercial specification available.
Iron -1 percent silicon investment casting.	Hitchener Manufacturing Co., Milford, N.H.	AMS5210
Nivco alloy sheet and bar. (23Ni - 1.1Zr - 1.8Ti-Co)	Westinghouse Research and Development Center Pittsburgh 35, Pa., and Westinghouse Materials Manufacturing Dept., Blairsville, Pa.	No commercial specification available.
Maraging steel bar and sheet (15Ni - 9Co - 5Mo - Fe)	Allegheny Ludlum Steel Corp.	Allegheny Ludlum Product Specification Almar 15

AISI Grade H-11 sheet .050" thick premium quality material . (5Cr - 1Mo - 1V - Fe)	Universal Cyclops Steel Corp., rerolled to 0.014 and 0.025" and heat treated by Westinghouse Research and Development Center, Pittsburgh 35, Pa.	AMS6437
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AISI Grade H-11 bar and forgings premium quality material. (5Cr - 1Mo - 1V - Fe)	Universal Cyclops Steel Corp.	AMS6487
--	----------------------------------	---------

Two of these materials, Nivco and H-11 thin-gage sheet, were not commercially available at the time this program was started. Consequently, sheet rolling technology was developed to produce limited quantities of both alloys for this program. A detailed account of the rolling procedures developed will be presented in the next quarterly report.

## 2. Sample Preparation

### a. Solid DC Test Ring

All the heat treatable alloys are machined to 0.020 inch oversize, heat treated, finish machined, and ground to size. The heat treatments used on the different alloys are listed later in this section.

### b. Rowland Ring Test Samples

The sheet materials were punched and blanked with precision dies to hold deburring to a minimum. After blanking, all laminations (except those of Supermendur, which were returned to Arnold Engineering Corp. for further processing) were degreased and insulated with one coat of aluminum-ortho-phosphate to prevent sticking during magnetic annealing or stress relief annealing. A second coat of aluminum-ortho-phosphate was applied after annealing as an interlaminar insulation.

All the finished cores are wound with insulated wire and tested. Wire selection depends on the test temperature and the driving voltages. A glass-served copper wire and nickel-coated copper insulated with Westinghouse 2554B insulation is used for tests to 1100°F and 100 volts. Above these limits Anaconda Wire and Cable's Anadur is used.

c. Mechanical and Physical Test Specimens

1.) Smooth-Bar Specimens

- a) Rough machine to 0.020 inch oversize
- b) Heat treatment when required
- c) Rough grind to 0.008 inch oversize
- d) Finish grind in 0.0002 inch steps to 0.001 inch oversize with coolant and polish with 180, 400, and 600 grit abrasive as necessary to achieve the specified finish and size.
- e) When specified, lap to the indicated finish.

2.) Notched-Bar Specimens

Follow the general procedure outlined above with the following exceptions:

- a) After heat-treatment, rough machine the notch to 0.010 oversize.
- b) Finish machine the notch to size with a carbide-tipped cutting tool which has been exactly ground to the notch contour.
- c) Finish and polish, using an appropriate string impregnated with abrasive for finishing the notch.

d. Specimen Heat Treatment

1.) H-11 Bar

Preheat the test bars to 1200°-1300°F, transfer to a hydrogen-gas atmosphere furnace and heat to 1850° ± 25°F. Hold at temperature for one hour and air quench to room temperature. Temper the parts three times in air at 1120°-1140°F for three, 1-1/2 hour, periods to achieve a final hardness of Rockwell C44 to C45.5.

2.) One Percent Silicon-Iron

Annealed per AMS 5210

3.) Westinghouse Nivco Alloy Bar

Heat to 1725° ± 15°F in an air atmosphere, hold at temperature 1 hour and water quench. Age-harden at 1225° ± 5°F for 25 hours in air to a minimum hardness of Rockwell C36.

4.) Hiperco 27 Bar

This material is ordered in the fully annealed condition.

5.) H-11 Sheet

H-11, 0.014 inch and 0.025 inch sheet is obtained in the annealed and cold rolled condition. The as-rolled sheet is punched and blanked, deburred, degreased and coated on both sides with aluminum-ortho-phosphate to prevent sticking during heat treatment. The heat treatment is conducted in a hydrogen-atmosphere furnace in which the atmosphere dewpoint is maintained at a maximum dewpoint of  $-40^{\circ}\text{F}$ . The punched laminations are held between two plates and preheated to  $1200^{\circ} - 1300^{\circ}\text{F}$  and then transferred to a hydrogen atmosphere furnace held at  $1850^{\circ} \pm 25^{\circ}\text{F}$ . Time at temperature is one hour and is followed by an air-blast quench. As quenched, the sheet has a hardness of Rockwell C60 (converted from Rockwell 15N). Hydrogen gas also acts as the protective gas during tempering at  $1120^{\circ} - 1140^{\circ}\text{F}$ . Three 1-1/2 hour temper cycles are required to bring the hardness to nominal Rockwell C45 and to effect complete transformation of austenite to martensite. After heat treatment, the rings are recoated on both sides with aluminum-ortho-phosphate and wound for magnetic test. Mechanical test sheet specimens are heat treated as above; however, the aluminum-ortho-phosphate treatments are omitted.

6.) Nivco Sheet

Nivco sheet, 0.014 inch and 0.025 inch thick, is solution heat treated in air, water quenched, pickled, and cold finished. Nivco sheet in the solution-annealed condition requires a 25-hour air age at  $1225^{\circ} \pm 5^{\circ}\text{F}$  to achieve a minimum hardness of Rockwell C38. Actual hardness achieved on trial samples approached Rockwell C43 (converted from Rockwell 15N).

A series of photographs showing typical examples of mechanical test specimens which have been completed are presented in Figures II-6 to II-14.

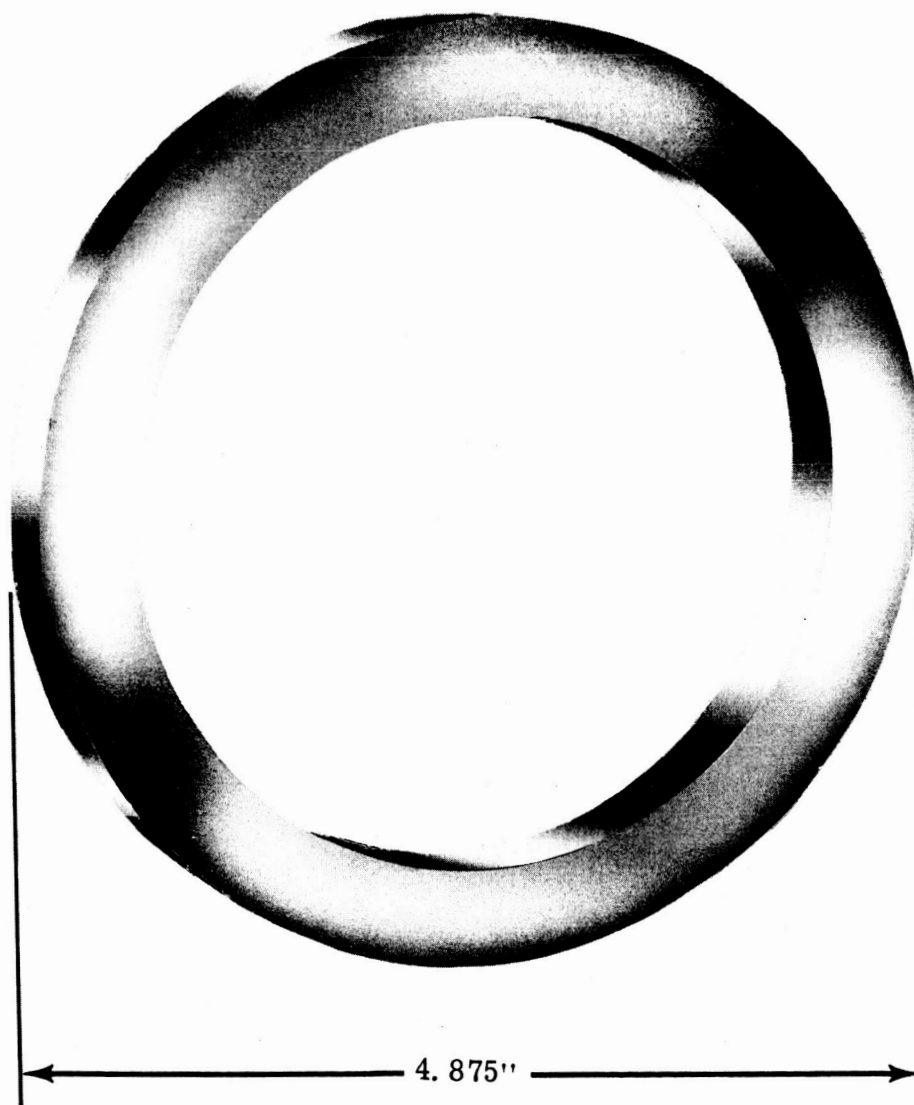
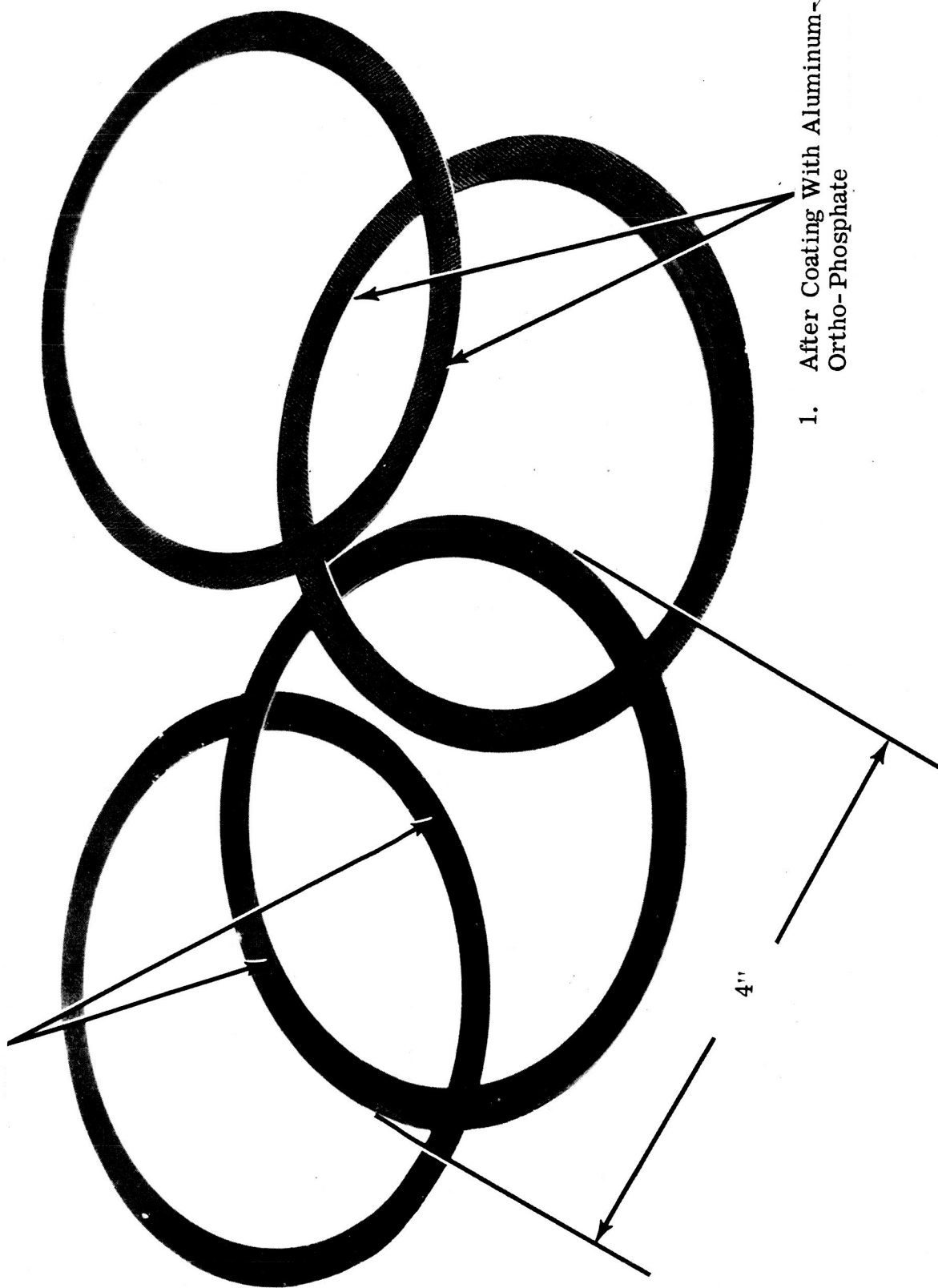


FIGURE II-6. Solid DC Magnetization Test Ring

WAED64.14E-21



2. As Punched



1. After Coating With Aluminum-Ortho-Phosphate

4"

FIGURE II-7. Laminated Magnetic Test Rings

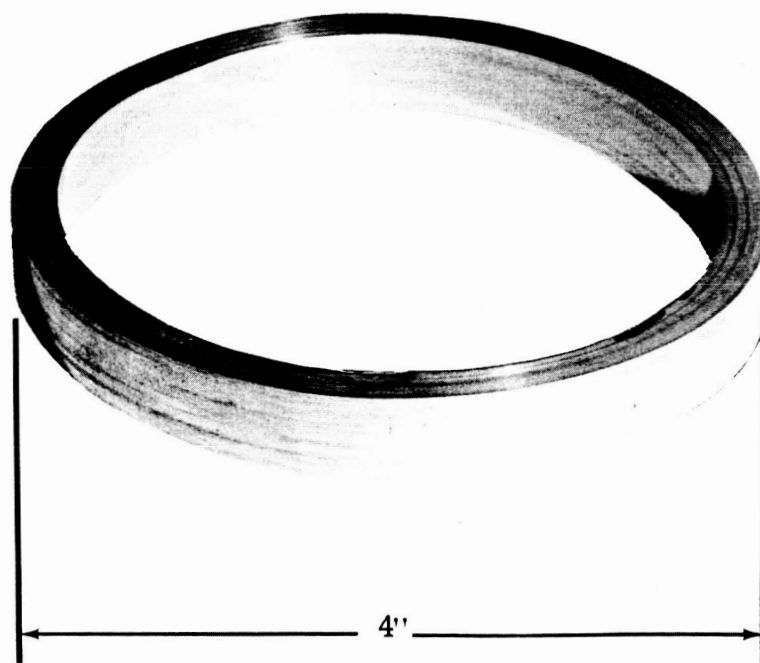


FIGURE II-8. Large Tape Wound Toroid

WAED64. 14E-23

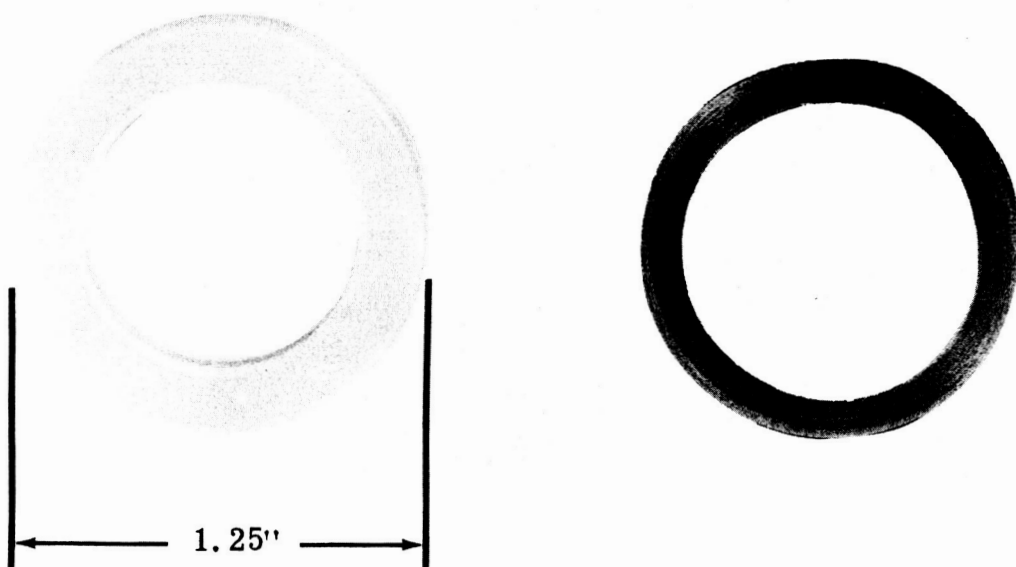


FIGURE II-9. Small Tape Wound Toroid and Protective Handling Case

WAED64.14E-24

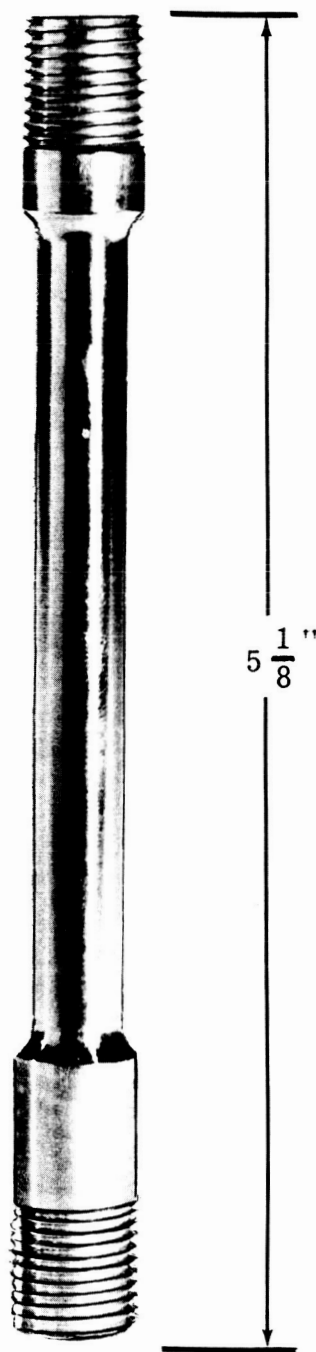


FIGURE II-10. Smooth Bar Tensile - Creep Specimen,  
0.357 inch Diameter

WAED64.14E-25

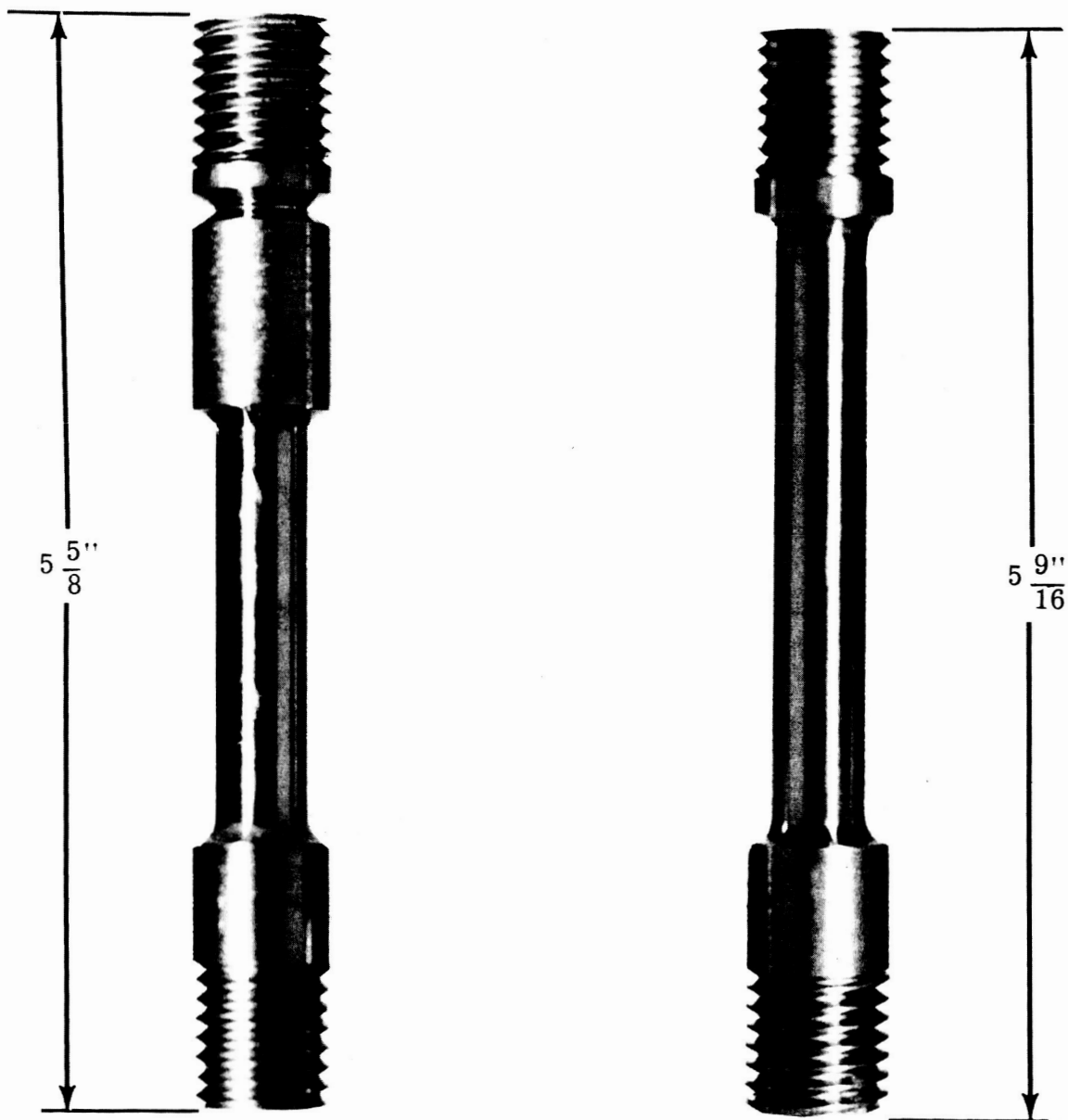


FIGURE II-11. Smooth Bar and Combination Smooth and Notched Bar  
Stress Rupture Specimens, 0.505 inch Diameter

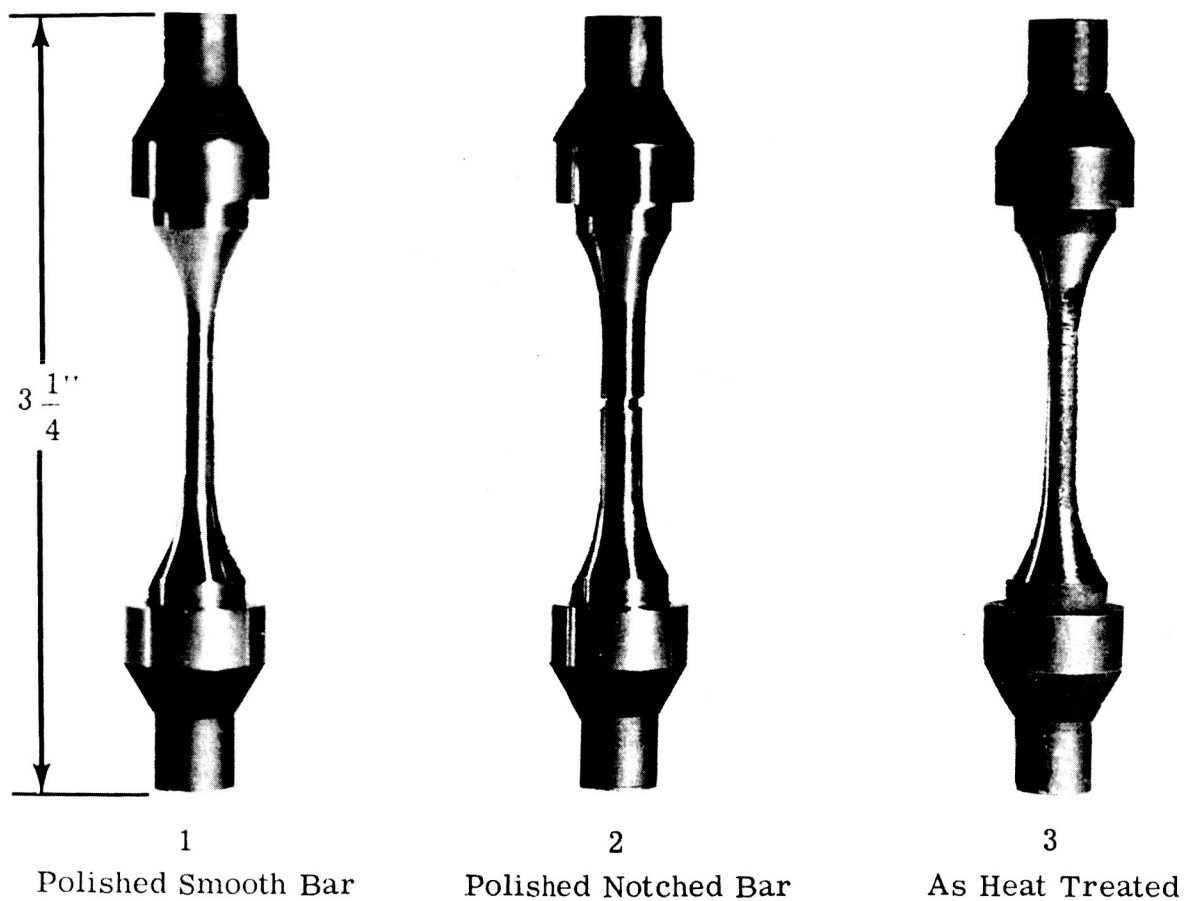


FIGURE II-12. Fatigue and Dynamic Creep Specimens,  
0.160 inch Nominal Diameter

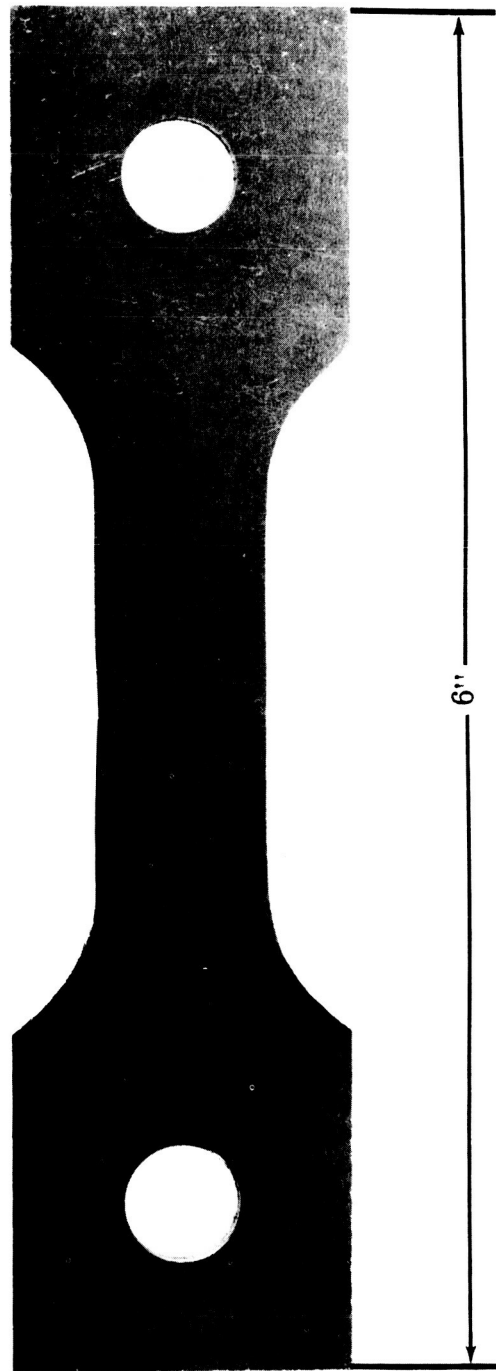


FIGURE II-13. Poisson's Ratio Specimen

WAED64. 14E-28

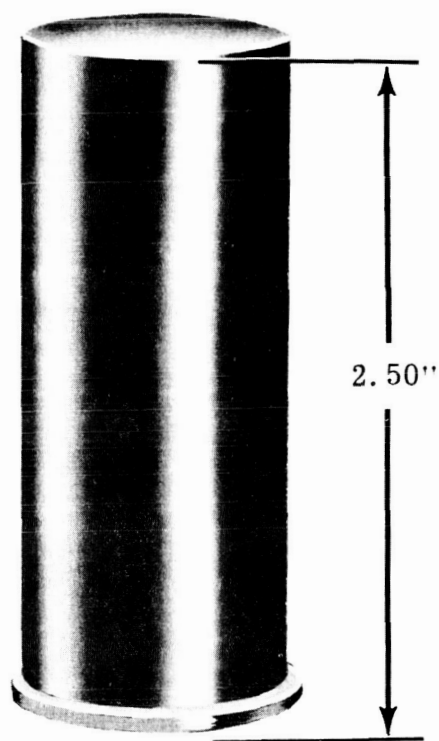


FIGURE II-14. Specific Heat Specimen

WAED64.14E-29



## E. MECHANICAL TEST RESULTS.

During the second quarter, the following were accomplished on the mechanical portion of the magnetics test program:

- 1) Room temperature Poisson's ratio measurements were made on a Hipercor 27 forging and 0.012 inch thick Cubex sheet.
- 2) Tensile tests were completed on the vacuum melted, forged Hipercor 27 bar material over the temperature range of 70°F to 1400°F.
- 3) Tensile tests were completed on AMS5210 (investment cast 1 percent silicon-iron) over the temperature range of 70°F to 1000°F.
- 4) Elevated temperature tensile tests were started on Nivco alloy forgings.
- 5) Room temperature and 800°F fatigue and dynamic creep tests were made on smooth and notched bar samples of AISI grade H-11.
- 6) Creep testing of the Hipercor 27 and Nivco alloy forged stock is in progress.

Testing was performed in accordance with the latest ASTM standards.

### 1. Discussion of Results

#### a. Poisson's Ratio (Cubex)

Poisson's ratio data for 0.012 inch thick Cubex are presented in Table II-4. Considerable scatter in the data had been expected because of unusually large normal grain size of the alloy. To determine the source and amount of the scatter, eight strain gages were mounted on each of two Poisson's ratio sheet specimens. Normally, only four strain gages are required for accurate strain measurements. The eight gages were arranged as shown in Figures II-15 and II-16. Note that the gages were placed in both the longitudinal and transverse directions on a single grain as well as across the grain boundaries. The Baldwin-Lima-Hamilton type A-7 gages were mounted on the sample with Duco Cement, and were chosen for their size, stability, and ease of mounting.

For accuracy, a Wiedeman Baldwin Mark B-20 testing machine was used in conjunction with a Budd digital strain indicator. Strains were recorded at numerous load increments with each set of readings made at constant load.

TABLE II-4. Poisson's Ratio of 0.012 Inch Thick Cubex  
at Room Temperature

Specimen No.	Both Gages on One Grain	Both Gages Across Grain Boundary	Maximum*	Minimum*
1	0.325	0.362	0.419	0.325
2	0.310	0.345	0.433	0.303
*These values were calculated without regard to the location of the strain gages as shown in Figures II-15 and 16.				

Reference: NAS3-4162

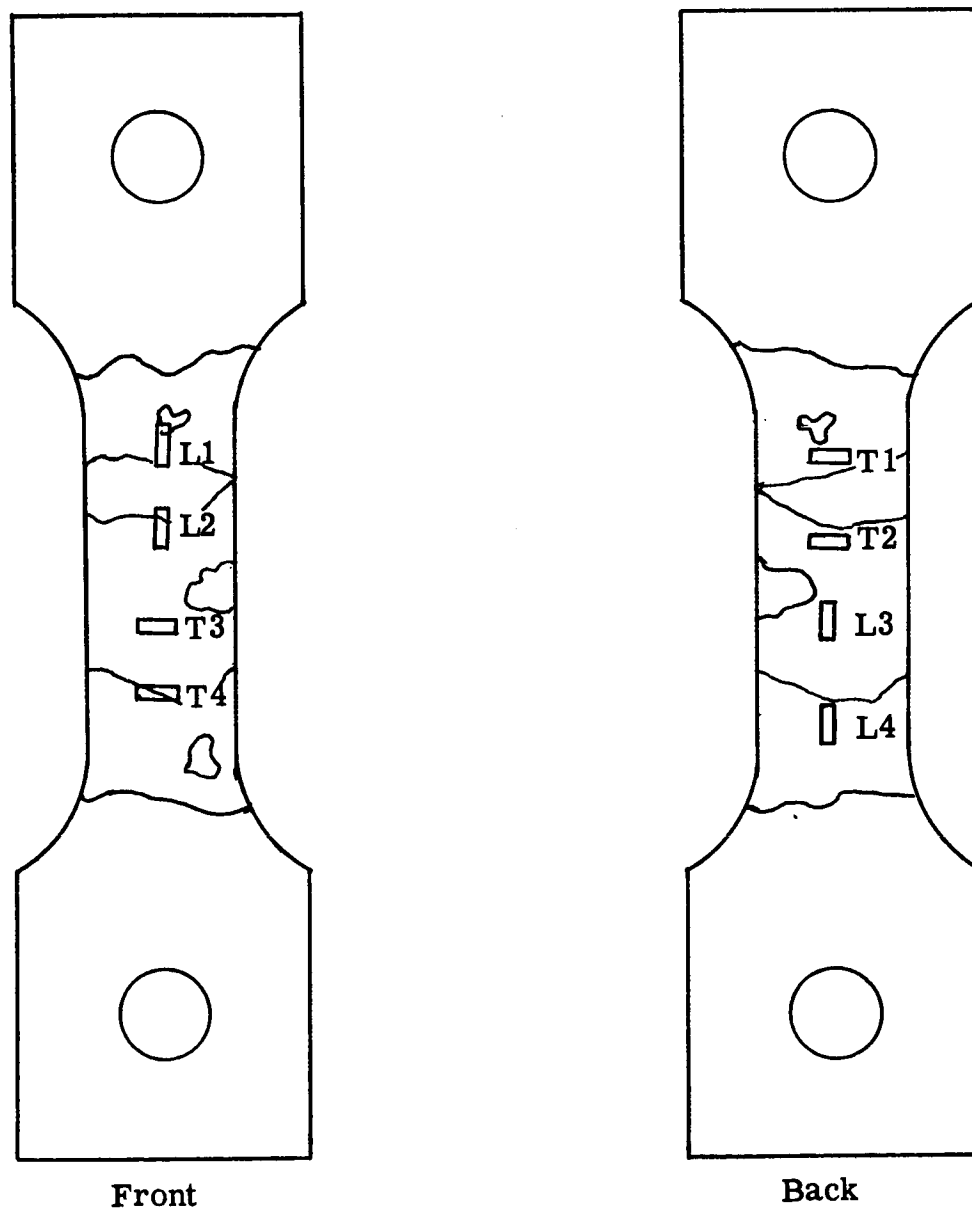


FIGURE II-15. Cubex Specimen No. 1, Showing Orientation of Gages With Reference to Cube Structure (Specimens Etched With Diversey 914)

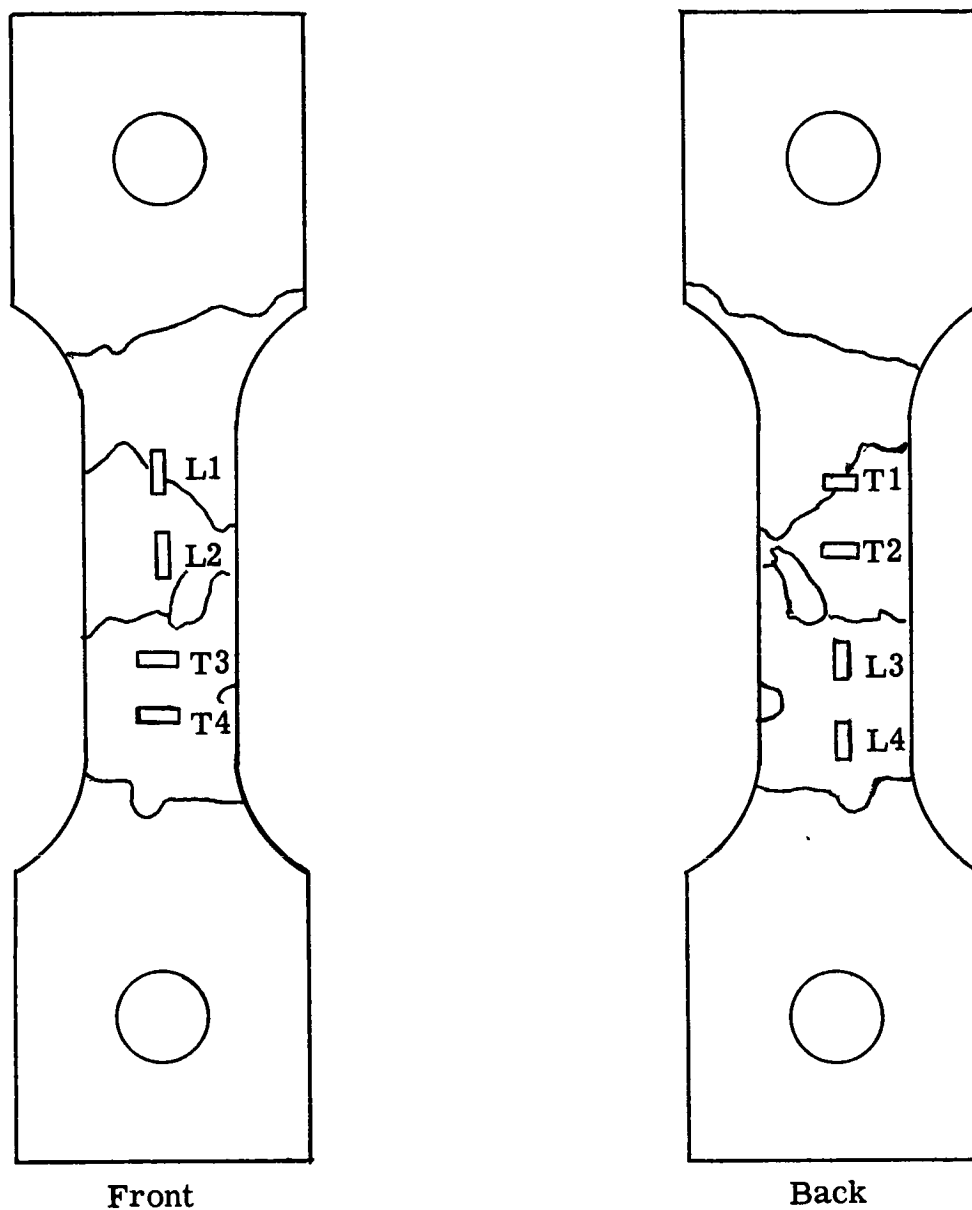


FIGURE II-16. Cubex Specimen No. 2, Showing Orientation of Gages With Reference to Cube Structure (Specimens Etched With Diversy 914)

The summary of data presented in Table II-4 shows that the measured values of Poisson's ratio are influenced by the location of the strain gages. Strains measured in a single grain differ considerably from strain measured across the boundaries between adjacent grains.

Published information on the measurement of Poisson's ratio for some materials notes an effect from applied stress on the measured value. No such effect was noted in the Cubex data.

b. Poisson's Ratio (Hiperco 27)

The Poisson's ratio data for vacuum melted forged Hiperco 27 are presented in Table II-5. Six average values were obtained on two separate specimens. Individual values were obtained for every 250 pounds of specimen load up to a stress of 48,450 psi. The Hiperco specimens (unlike the Cubex samples) were machined from solid bar stock and were 0.125 inch thick. Two longitudinal and two transverse strain gages were cemented to each of the sample as with Cubex. Hiperco 27 is a fine grained material and was not expected to cause difficulties in the measurement of Poisson's ratio as shown in Figures II-17 through II-22.

c. Tensile Properties (Hiperco 27)

Hiperco 27 tensile properties, elongation, reduction in area, and modulus of elasticity measured over the range of room temperature to 1400°F, are presented as data plots in Figures II-22 to II-25. A low indicated yield strength was noted in one room temperature specimen of Hiperco 27. The yield strength of this sample was a yield point rather than 0.20 percent off-set yield strength. No explanation of the appearance of the yield point can be offered. The short-time elevated temperature tensile data appears to show no effect of test atmosphere on the properties of Hiperco 27. One of the 1000°F specimens and both the 500°F and 1400°F specimens were tested in argon to check for possible adverse effects of the air atmosphere on the data. Creep tests were also run at 1400°F on Hiperco 27 in both argon and air to further check for possible adverse effects of the ambient atmosphere. No significant differences in creep rate or tensile properties were noted between the air or argon tested samples, tested to 700 hours.

An expected drop in the elevated temperature ductility of Hiperco 27 (Figure II-24) occurs in the temperature range between 700°F and 1400°F. This drop is observed in nearly all materials and marks the temperature range at which vacancies are generated and

TABLE II-5. Poisson's Ratio of Hiperco 27 at Room Temperature

Specimen No.	Run No.	Width (Inches)	Thickness (Inches)	Poisson's Ratio *	Average Poisson's Ratio
1	1	0. 749	0. 124	0. 305	0. 320
	2			0. 327	
	3			0. 329	
2	1	0. 749	0. 124	0. 330	0. 335
	2			0. 348	
	3			0. 328	

\*These data were obtained from the stress strain plots in Figures II-2 to II-7 where  $\mu = e_L/e$ .  $\mu$  = Poisson's Ratio  
 $e_L/e$  is the ratio of unit lateral strain to unit axial strain below the elastic limit.

Reference: NAS3-4162

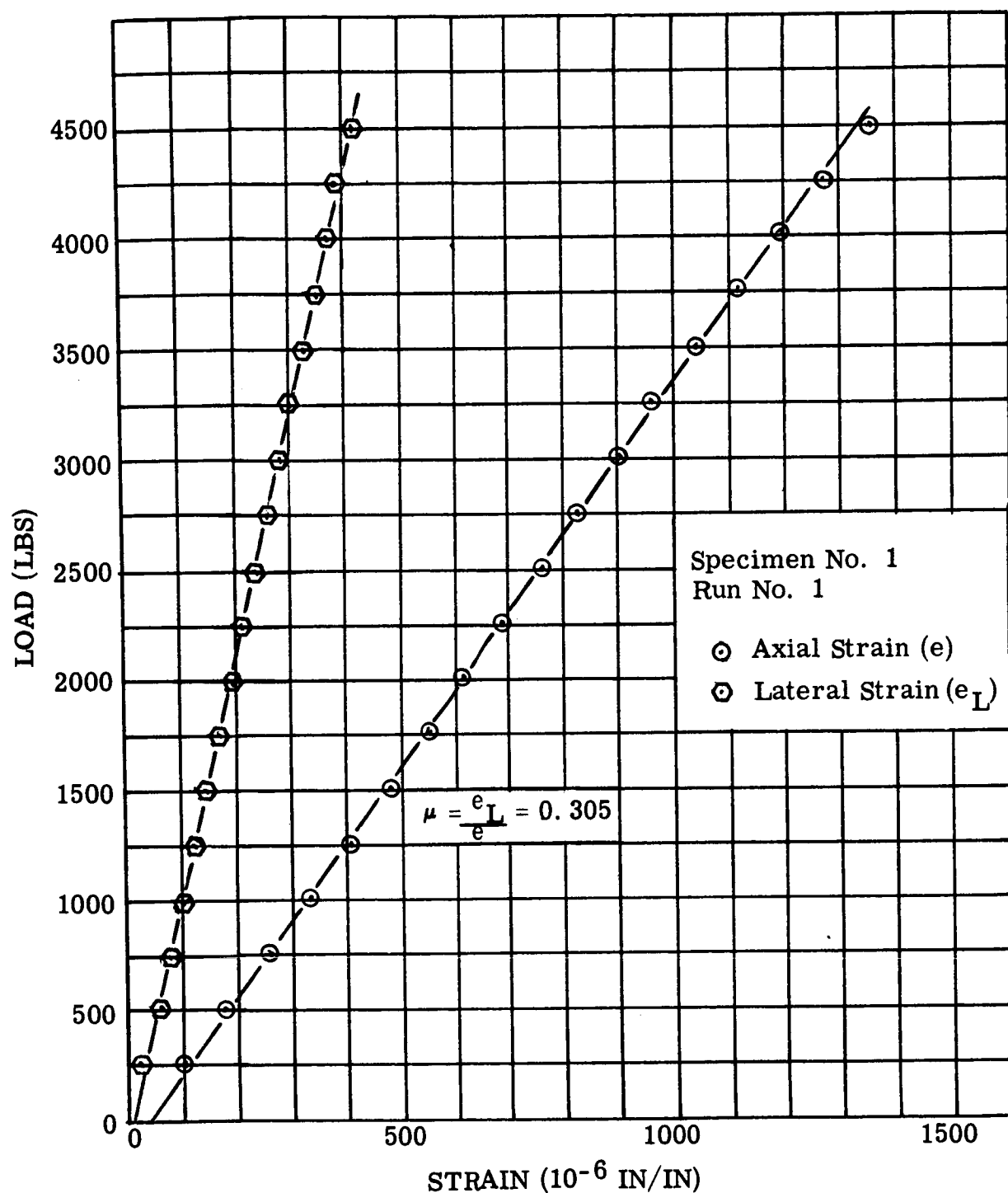


FIGURE II-17. Elastic Strain Versus Load For Hiperco 27 Alloy  
Room Temperature Test (Reference: NAS3-4162)

Poisson's Ratio - Hiperco 27

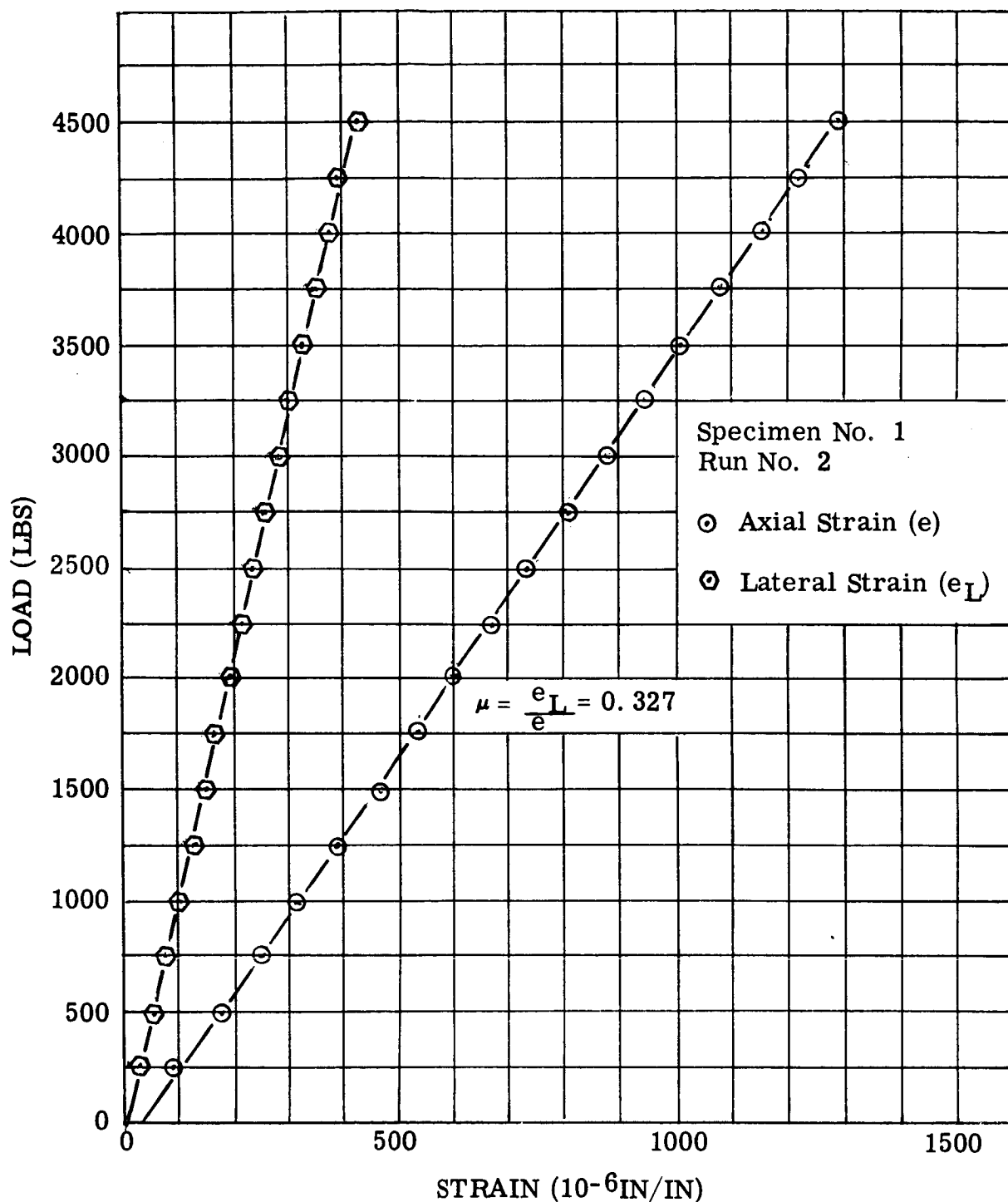
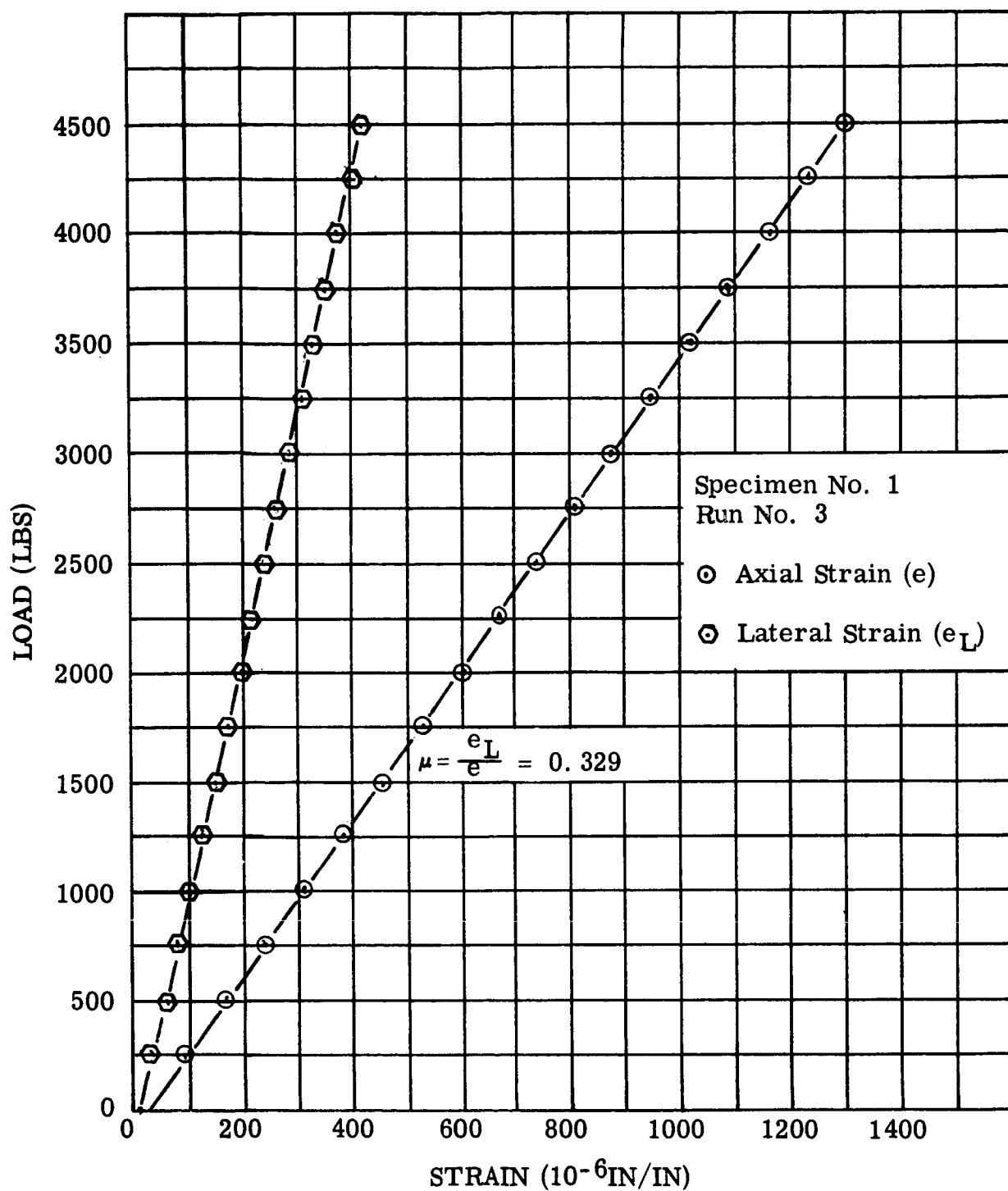


FIGURE II-18. Elastic Strain Versus Load For Hipercó 27 Alloy  
Room Temperature Test (Reference: NAS3-4162)

Load vs. Strain - Hipercó 27





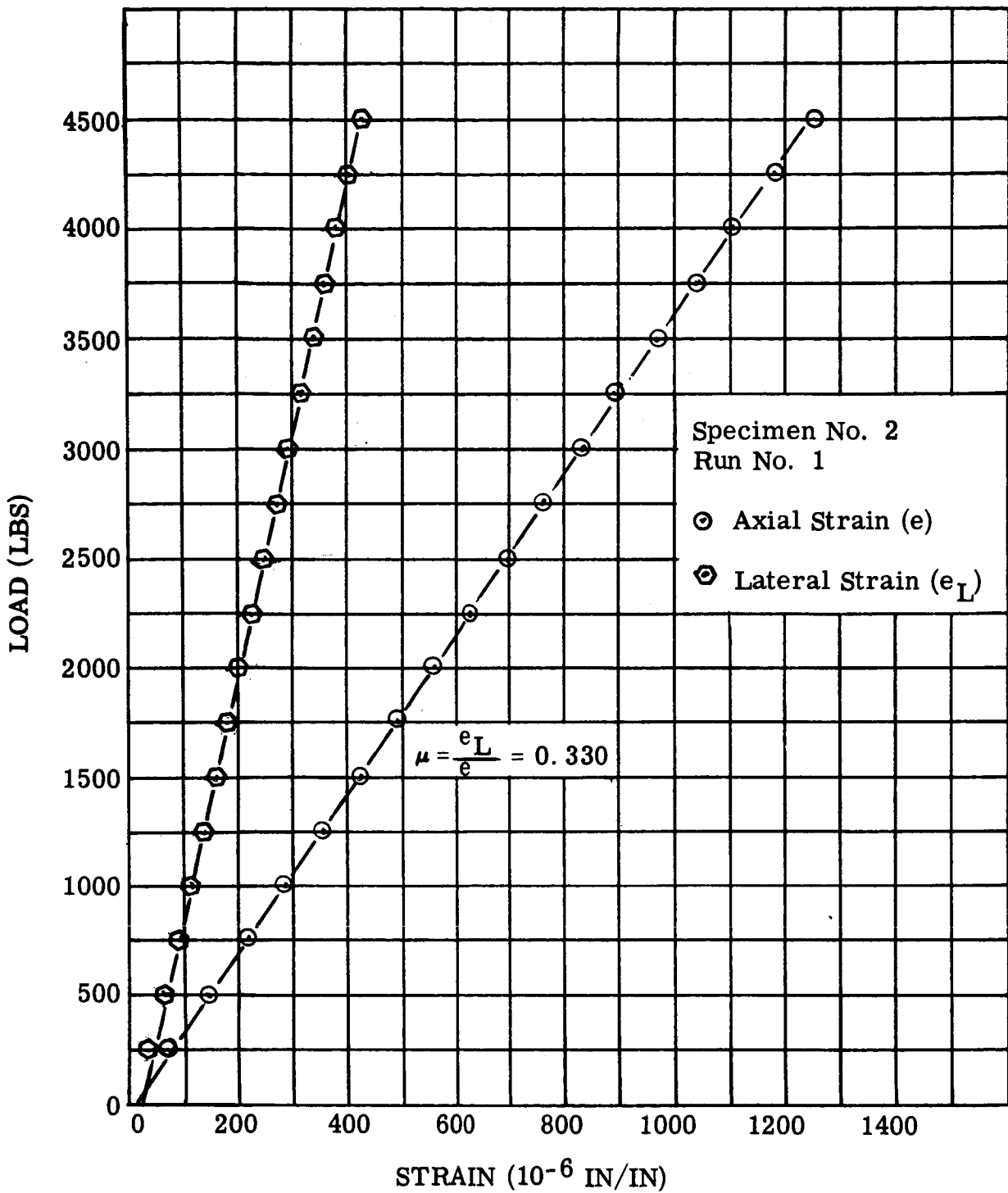


FIGURE II-20. Elastic Strain Versus Load For Hiperco 27 Alloy  
Room Temperature Test (Reference: NAS3-4162)

Poisson's Ratio - Hiperco 27

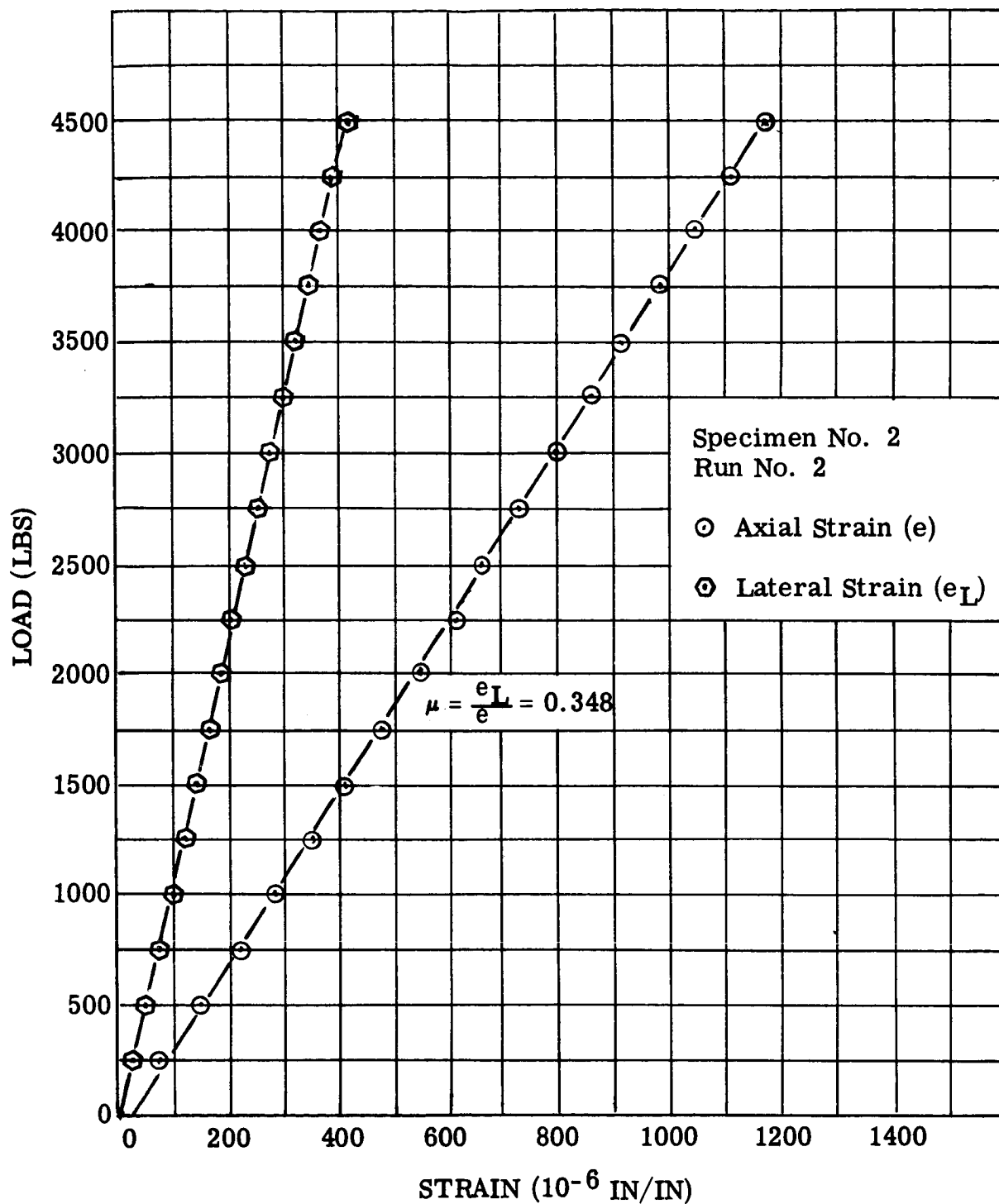


FIGURE II-21. Elastic Strain Versus Load For Hiperco 27 Alloy  
Room Temperature Test (Reference: NAS3-4162)

Poisson's Ratio - Hiperco 27

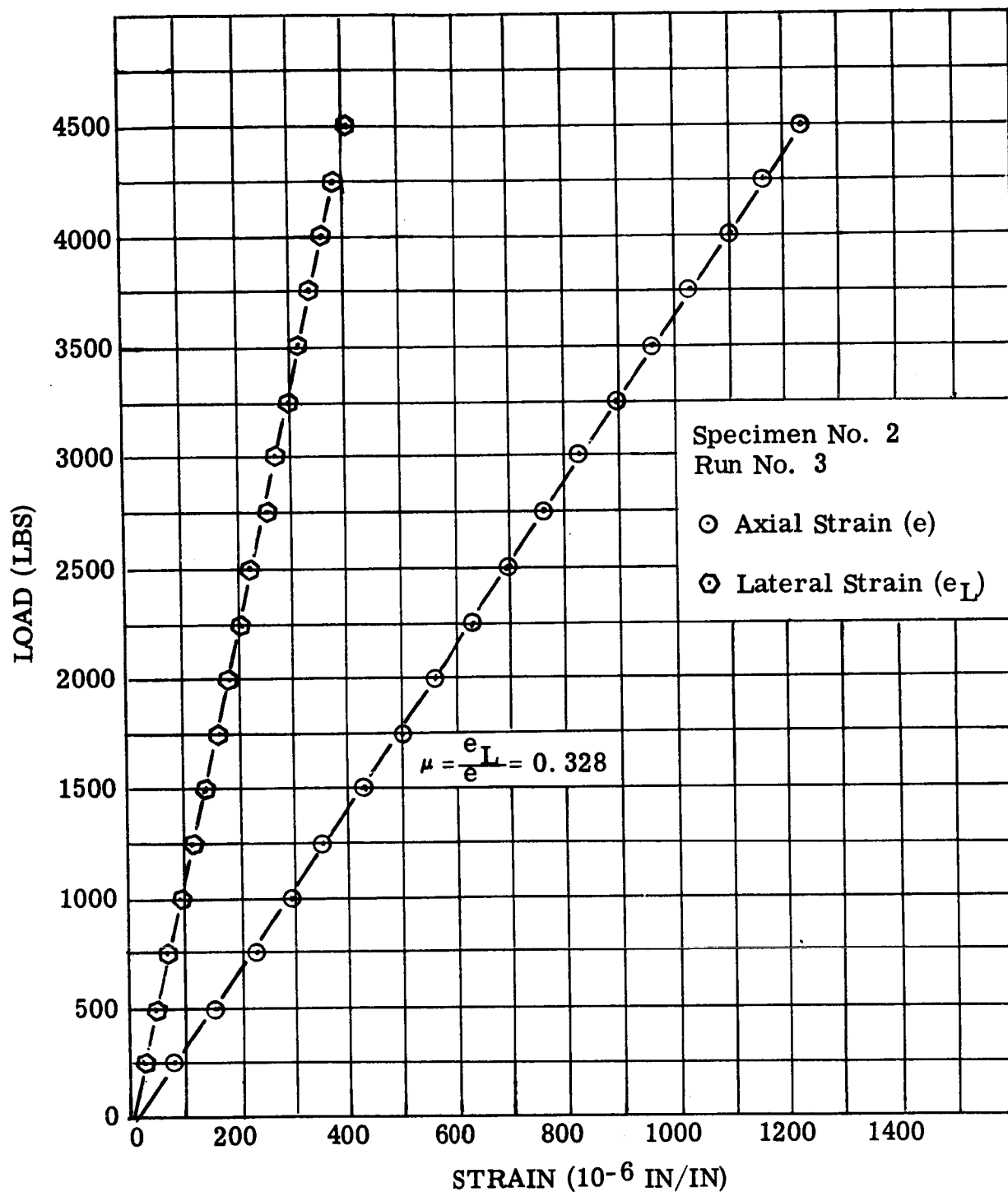


FIGURE II-22. Elastic Load Versus Strain For Hipercó 27 Alloy  
Room Temperature Test (Reference: NAS3-4162)

Poisson's Ratio - Hipercó 27

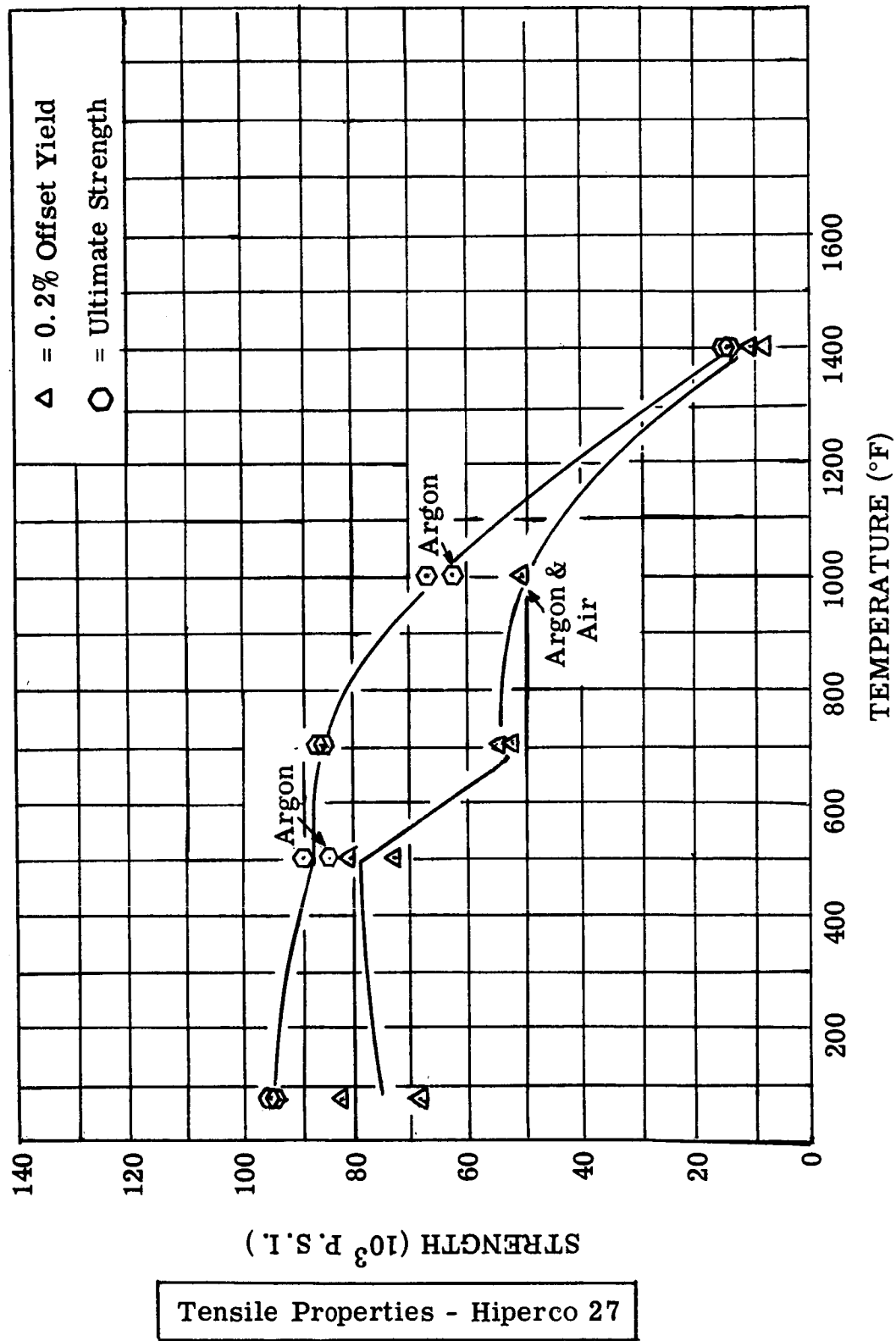


FIGURE II-23. Room and Elevated Temperature Yield and Tensile Strengths of Vacuum Melted and Forged Hipercro 27 Alloy Air and Argon Tests. Data Taken From Two Heats of Material (Reference: NAS3-4162)

# Tensile Ductility - Hiperco 27

WAED64. 14E-43

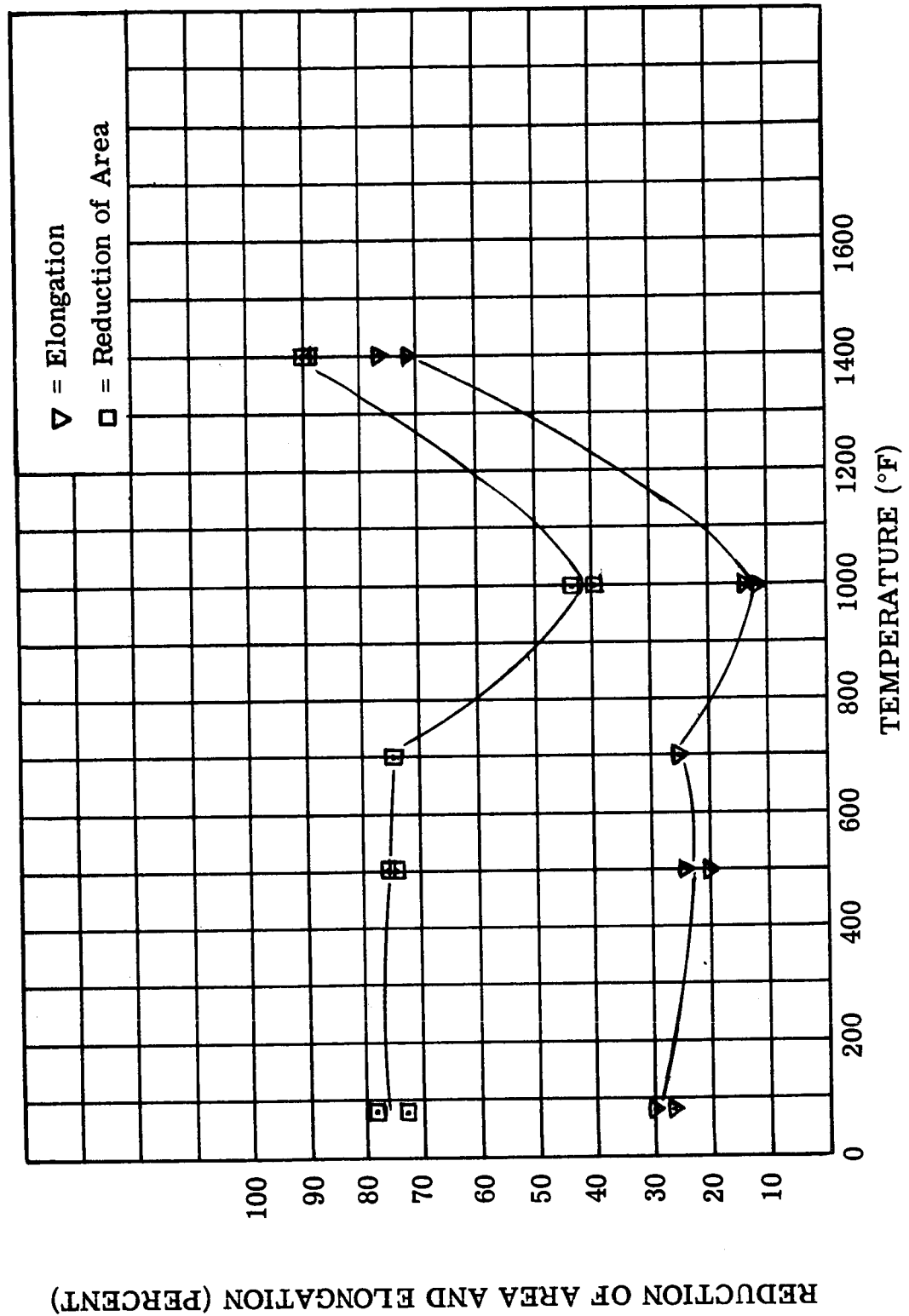


FIGURE II-24. Room and Elevated Temperature Tensile Elongations and Reduction of Areas for Vacuum Melted and Forged Hiperco 27 Alloy. Data Taken From Two Heats of Material (Reference: NAS3-4162)

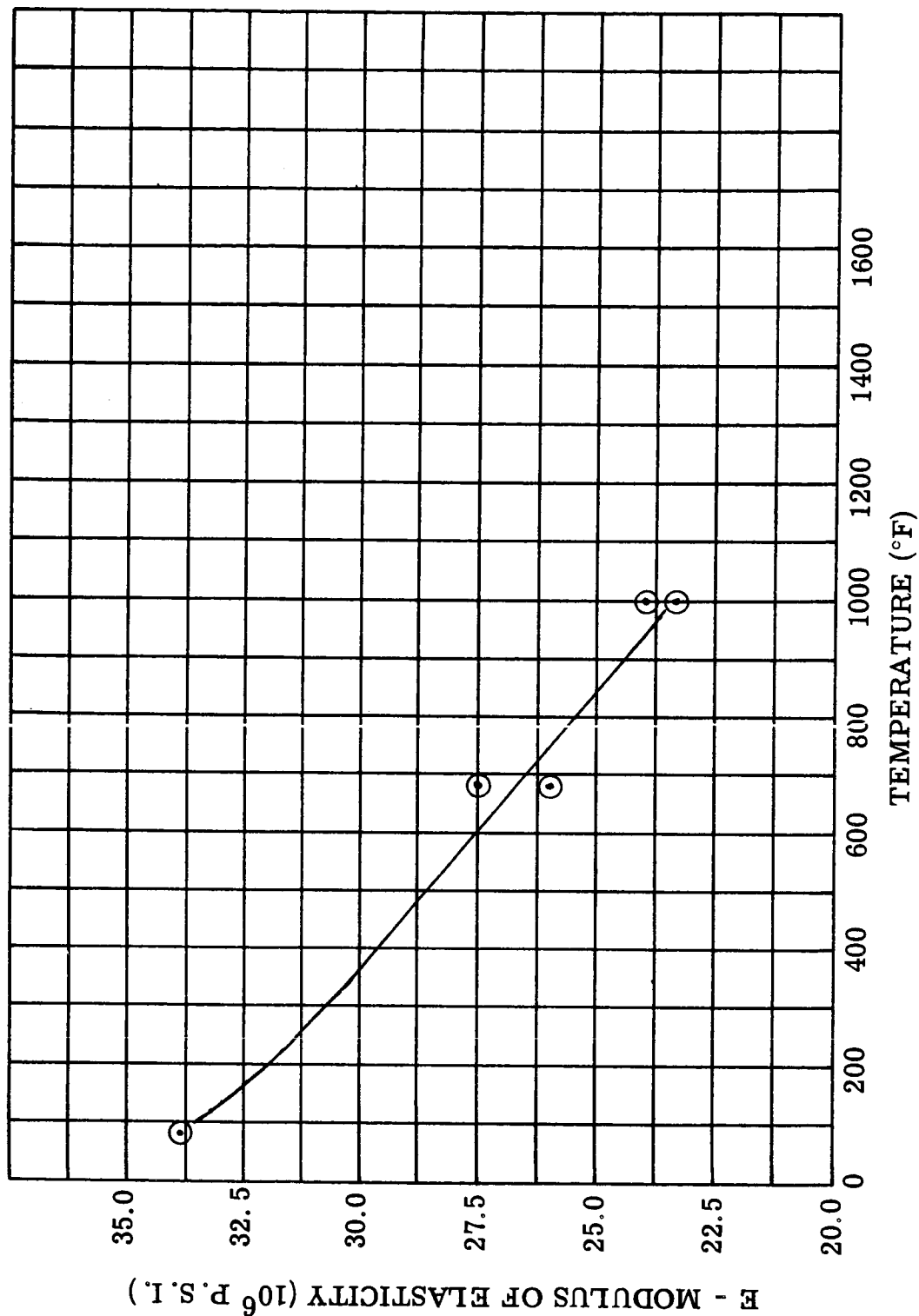


FIGURE II-25. Room and Elevated Temperature Modulus of Elasticity of Vacuum Melted and Forged Hipercó 27 (Reference: NAS3-4162)

Youngs Modulus - Hipercó 27

WAED64.14E-44

move to grain boundaries but are not subsequently annihilated by recrystallization. The mode of fracture changes from transgranular to intergranular and back to transgranular over this temperature range.

d. Tensile Properties (1 Percent Silicon-Iron)

The tensile properties of AMS5210 are presented in Table II-6 and Figures II-26 and II-27. The cast bars were annealed per AMS5210 before testing. No clarification of the tensile data is required for this material. The expected ductility minimum of AMS5210 occurs at 500°F<sup>10</sup>.

e. Creep

Both Hiperco 27 and Nivco Alloy forgings are under test. On the basis of tests completed, Hiperco 27 lacks the strength required to determine 10,000 hour strength at 1400°F. The strain-versus-time curves for two of the Hiperco 27 specimens are plotted in Figure II-28. A number of individual test specimens will be stressed for periods of time not exceeding 1000 hours to determine those stresses which will produce 0.2 percent and 0.4 percent extension in 10,000 hours. The stresses will be selected to yield 0.4 percent true strain in approximately 100, 500, 750, and 1000 hours. The extension time curves produced in the above tests will be used to determine a time for 0.2 percent strain.

All creep tests will be made in air atmosphere unless degradation of the specimens is observed. Such degradation will be determined by examination of the test specimens.

Creep specimens of Hiperco 27 will also be tested in vacuum ( $10^{-6}$  torr) for a comparison with other atmospheres. When a protective atmosphere is preferred to the normal ambient atmosphere, the Hiperco 27 tests will be run in flooded-argon capsules.

f. Fatigue

Fourteen 800°F fatigue tests were completed on the H-11 material during the second quarter. An unfortunate situation occurred during the heat treating of the rough machined samples which invalidates the smooth-bar fatigue data obtained so far. All of the rough-machined

<sup>10</sup> Reid, B. J., Greenwood, J. N., "Intergranular Cavitation in Stressed Copper Nickel Alloys", AIME Transactions, Vol. 212, No. 4, p. 503, Aug. 1958.



samples which invalidates the smooth-bar fatigue data obtained so far. All of the rough-machined samples were accidentally preheated in a strongly decarburizing atmosphere. Approximately 0.005 inch to 0.010 inch of decarburization is normal for this material so that 0.020 inch of stock was left for heat treatment. The severity of decarburization was not noticed after heat treatment because the heat treatment hardness control piece was wet ground until a uniform hardness of Rockwell C45 was observed. Since the amount of material removed from the control piece was not measured, it was not evident that severe decarburization had occurred on the entire specimen. Metallographic examination and microhardness readings ultimately revealed between 0.001 inch and 0.003 inch of decarburization on the finished smooth-bar samples. Since the notches are not cut until after heat treatment, the notched fatigue data should be representative. New smooth-bar specimens were rough machined, heat treated, hardness checked for decarburization and finish machined by six days from the time the initial difficulty was observed.

The real effect of the decarburized layer remains in question since these data at a large number of cycles agree with data obtained by another investigator on H-11 material heat treated to a much higher hardness (Rockwell C52). The fatigue data will be presented in the next quarterly after additional analysis of present data and tests now underway have been made.

TABLE II-6. Room and Elevated Temperature Properties of AMS5210 (1Si-Fe)

Spec. No.	Diameter (Inches)	Test Temp (°F)	Hardness (Rockwell B)	0.02 Percent Offset Yield Str. (psi)	0.20 Percent Offset Yield Str. (psi)	Ultimate Strength (psi)	Elongation in 1" (%)	Reduction of Area (%)
1	0.252	RT	64, 65, 66	25450	28450	55200	36.1	56.1
2	0.249	RT	65, 66, 66	25850	29450	53200	38.3	65.1
3	0.250	500	62, 63, 65	19550	25250	60700	21.5	+
4	0.248	500	65, 66, 67	22750	26200	62100	22.6	44.3
5	0.251	800	64, 64, 66	20350	23150	41050	36.7	61.0
6	0.251	800	62, 63, 64	19550	22700	41000	36.7	74.5
7	0.248	1000	62, 63, 64	13150	15100	20800	53.6	80.7
8	0.249	1000	62, 63, 63	14100	15950	20400	63.0	85.2

+ Non uniform fracture area, % reduction not calculated.

Specimens were tested with strain rates as follows:

0.005 in/in/min to yield

0.050 in/in/min to failure

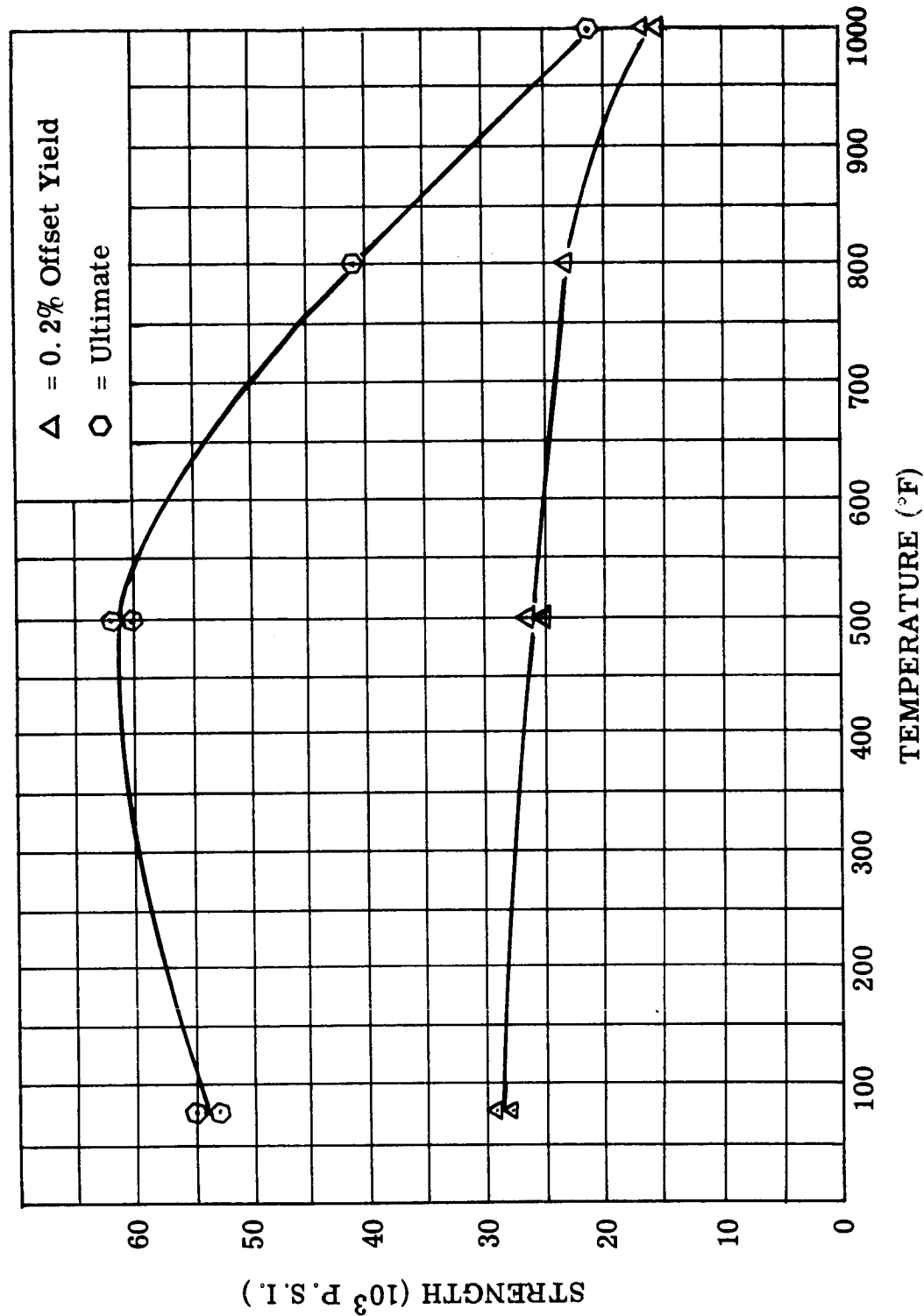


FIGURE II-26. Room and Elevated Temperature Tensile Strength Properties in Air AMS 5210 (1 Si-Fe) (Reference: NAS3-4162)

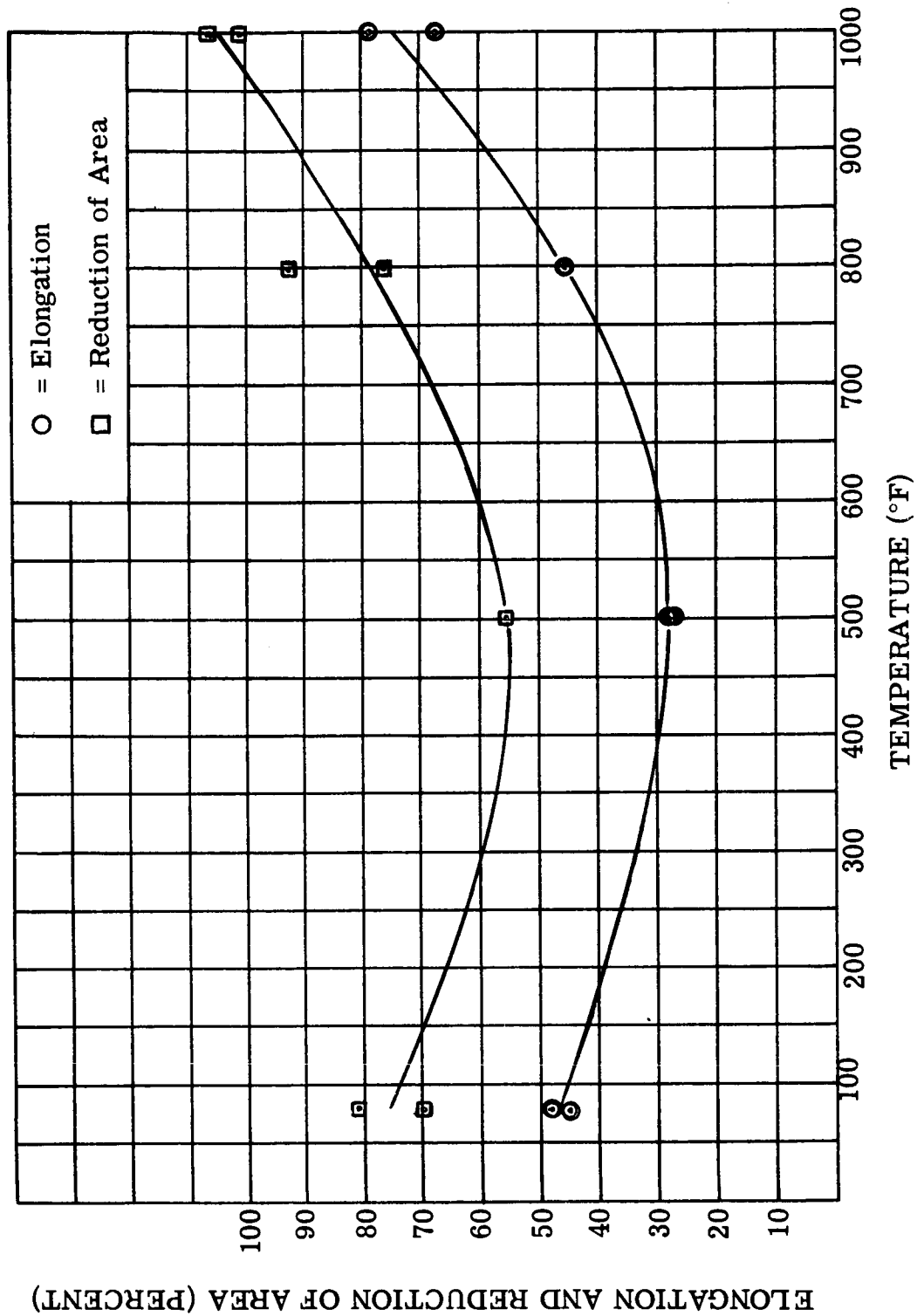


FIGURE II-27. Room and Elevated Temperature Tensile Ductilities of AMS5210  
(1 Si-Fe) (Reference: NAS3-4162)

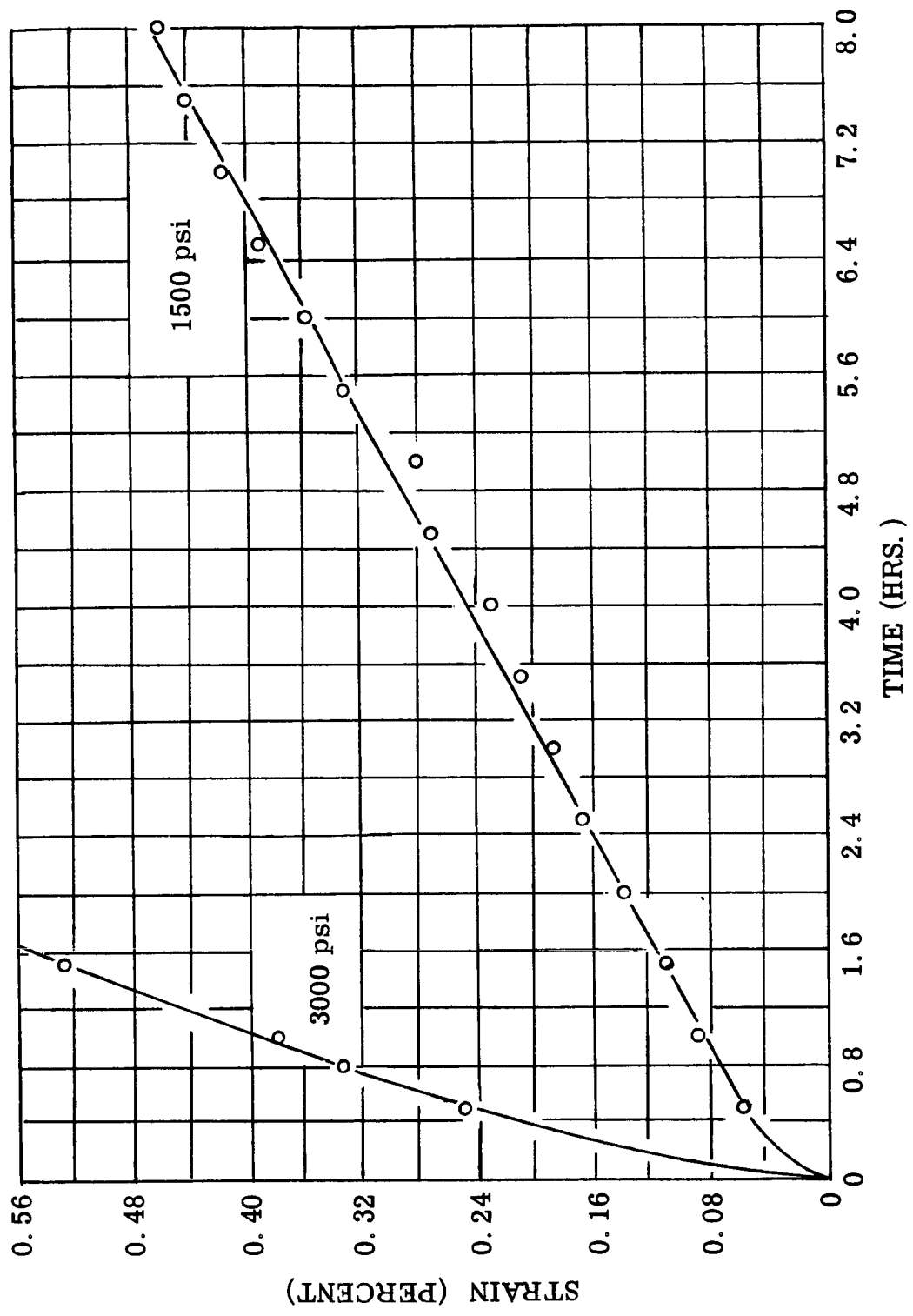


FIGURE II-28. Percent Strain For Hipercro 27 as a Function of Time at 1400°F in Argon  
(Reference: NAS3-4162)

Creep Rates - Hipercro 27

## SECTION III

### CONDUCTORS

#### A. INTRODUCTION.

Applications for elevated-temperature conductors may vary widely. In some instances the conductors may be required to withstand the temperature of operation and accompanying stress conditions for a relatively short time, while in other applications, such as in direct energy conversion, operational conditions may require long life and recycling capability. Further, environmental conditions may vary widely to include air, vacuum, inert gas, liquid and vapor alkali metals.

Ideally, it would be desirable that a conductor for high temperature use (500-1600°F) have the following characteristics:

1. As high an electrical conductivity as possible.
2. A low temperature coefficient of resistivity to minimize increase in resistivity at elevated temperatures.
3. Maximum resistance to oxidation and corrosion so that long time exposure does not increase the electrical resistivity.
4. Acceptable mechanical properties at room temperature and at operating temperature (yield strength, ductility, creep and rupture properties).
5. Acceptable fabrication characteristics, not only for the production of wire, but for the forming, and joining necessary in making the required final assembly.
6. Other desirable characteristics would include a magnetic permeability approaching 1.00, low vapor pressure (for vacuum applications), and hopefully, reasonable cost and availability.

**B. SUMMARY OF EFFORT IN THIS QUARTER .**

The conductor program during the past quarter resulted in final selection of the conductor materials. The following electrical conductors will be evaluated:

1. Austenitic stainless steel clad zirconium copper.
2. Austenitic stainless steel clad silver.
3. Inconel clad dispersion strengthened copper with a columbium barrier.
4. Inconel clad silver.
5. Nickel clad copper.
6. Thorium oxide dispersion strengthened nickel.
7. Beryllium oxide dispersion strengthened copper.

The test-program plan was completed and the method for endurance testing defined.

Two conductor materials (CuFo copper and nickel-clad copper) are being prepared for testing.

### C. DISCUSSION.

Two changes have occurred in the conductor program since the previous quarterly report. Molybdenum clad copper is not available at anything approaching a reasonable price and is considered a development speculation. Austenitic stainless clad silver will be evaluated as an alternate. DuPont will not be able to provide alumina dispersion-strengthened copper, as had been promised, since the material is still under research evaluation. Sufficient redraw rod was received from Handy and Harman to evaluate CuFo (Beryllia dispersion-strengthened copper) in both bare and clad conditions. The drawn "DS" copper wire and nickel clad copper series were received in February. The balance of the wires are to be delivered over the period of March 1 - April 15, 1964. Preliminary creep, tensile, and other thermophysical data from Handy and Harman and Sylvania on the CuFo (approximately 1% BeO) dispersion-hardened copper are presented in Table III-1 and Figures III-1 to III-3.

A comparison of the 750°F smooth-bar stress rupture properties of CuFo with the 600°F and 800°F stress-rupture properties of standard age-hardening beryllium copper No. 10 (2.5 percent cobalt, 0.5 percent beryllium, balance copper) is shown in Figure III-3. Beryllium copper No. 10 is considered the best high-strength, high-conductivity copper-base alloy currently available. Note that the dispersion strengthened material possesses a stress-rupture strength for 1000 hours and 750°F which has a factor of four better than that observed in fully hardened beryllium copper No. 10 at 800°F temperature. One longitudinal and one transverse micrograph of CuFo are shown in Figure III-4. The dispersion strengthened alloy (CuFo) also exhibits better electrical conductivity than the precipitation hardened beryllium copper alloy. The rupture ductility of both materials is low, on the order of 1 to 3 percent. It is not possible to estimate the design significance of the low rupture ductility at this time, however, CuFo appears to be a new and interesting high-temperature engineering material.



**TABLE III-1. Basic Product Data on CuFo From Handy and Harman**

Ultimate tensile strength at room temperature	80, 000-90, 000 psi	
0.2 percent offset yield strength at room temp.	75, 000-85, 000 psi	
Percent elongation	6-8	
Percent reduction of area at fracture	40-50	
Modulus of Elasticity	18.7 x 10 <sup>-6</sup> psi	
Electrical Conductivity (Percent of copper)	85	

Initial Room Temperature Test Data From Sylvania Electric Products Corporation After Drawing		
<u>Final Wire Diameter</u>	0.1006 inch	0.0401 inch
<u>Mechanical Properties</u>		
As hard drawn		
Percent elongation	7.0	1.5
Tensile strength	95, 000 psi	91, 000 psi
As stress relieved (1300°F, 1.5 hr in hydrogen)		
Percent elongation	12.0	8.0
Yield strength	67, 500 psi	69, 000 psi
Tensile strength	79, 000 psi	79, 000 psi
<u>Electrical Resistance (Ohm-cm)</u>		1.96(10) <sup>-6</sup>

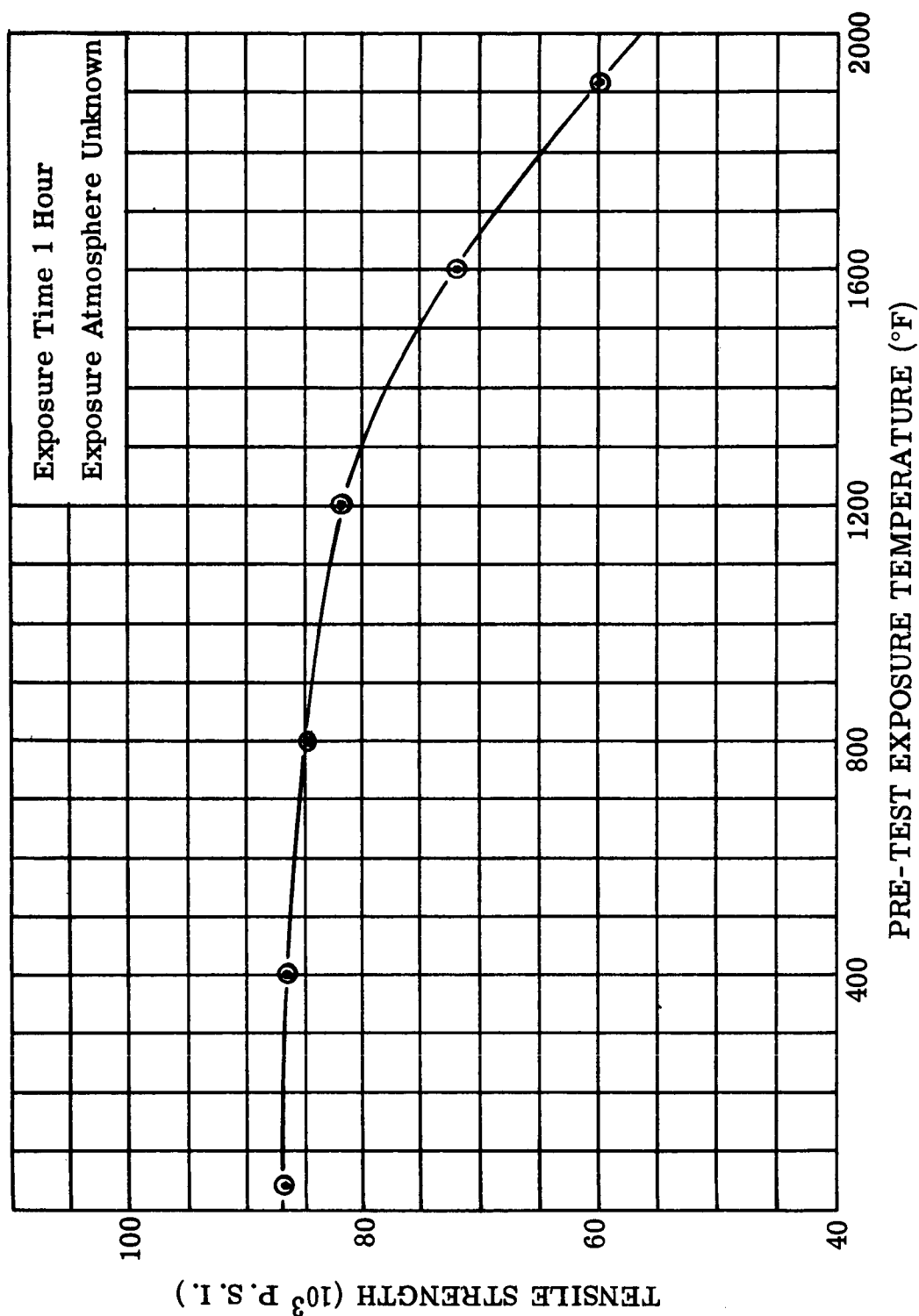


FIGURE III-1. Room Temperature Tensile Strength of Handy and Harman's CuFo After a One Hour Exposure at the Indicated Temperature.  
(Reference: Handy and Harman Product Literature, 1964)

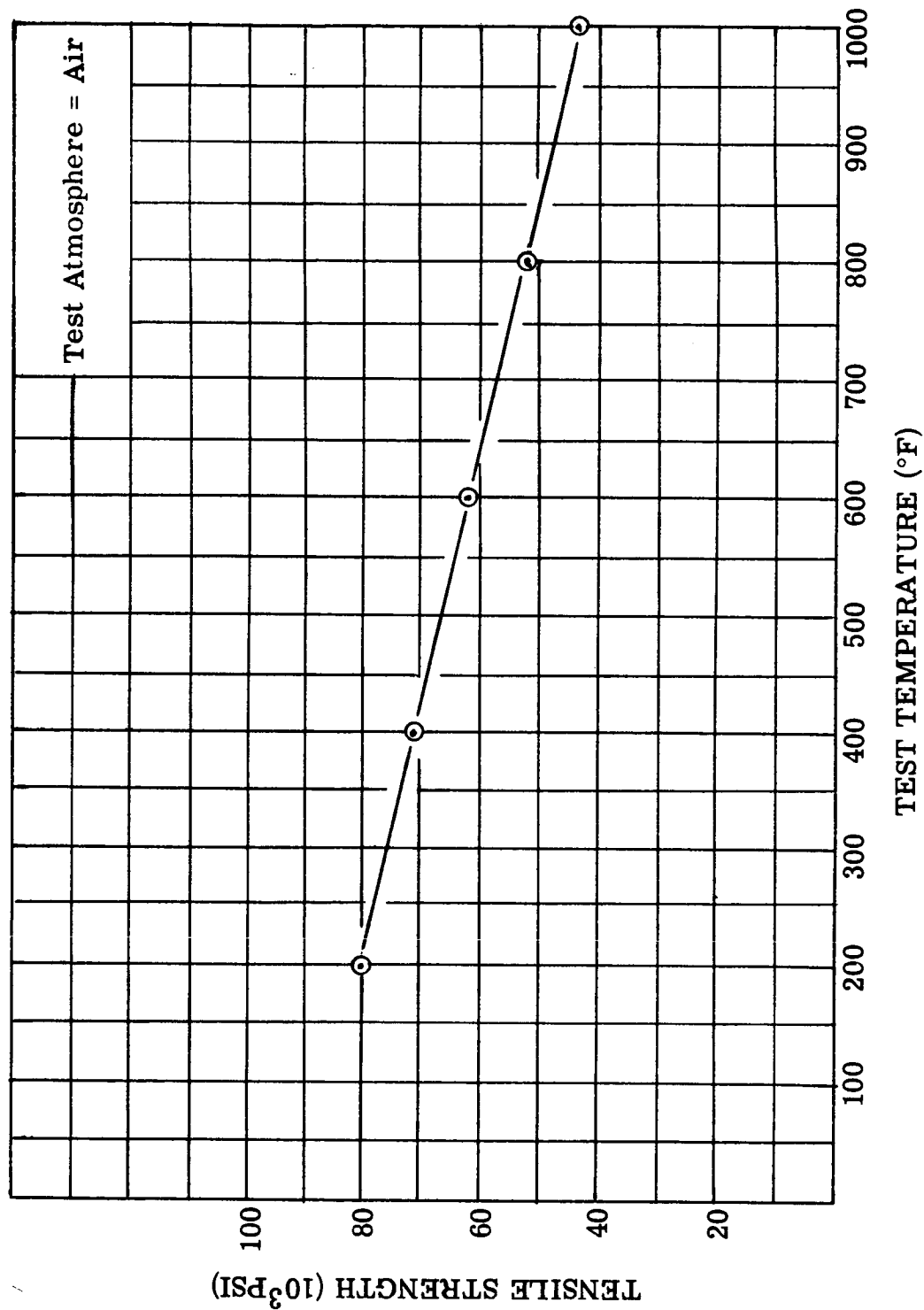
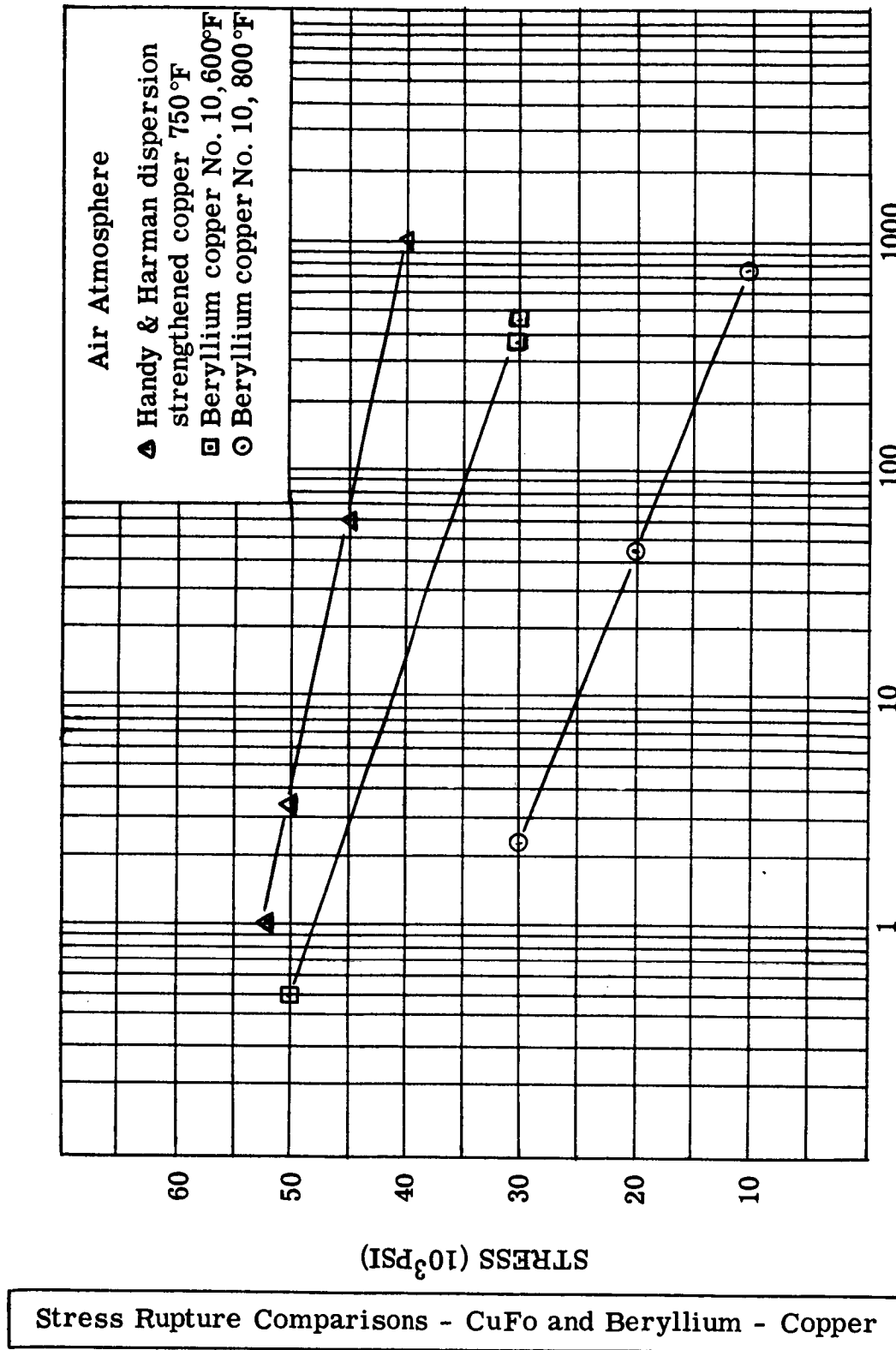


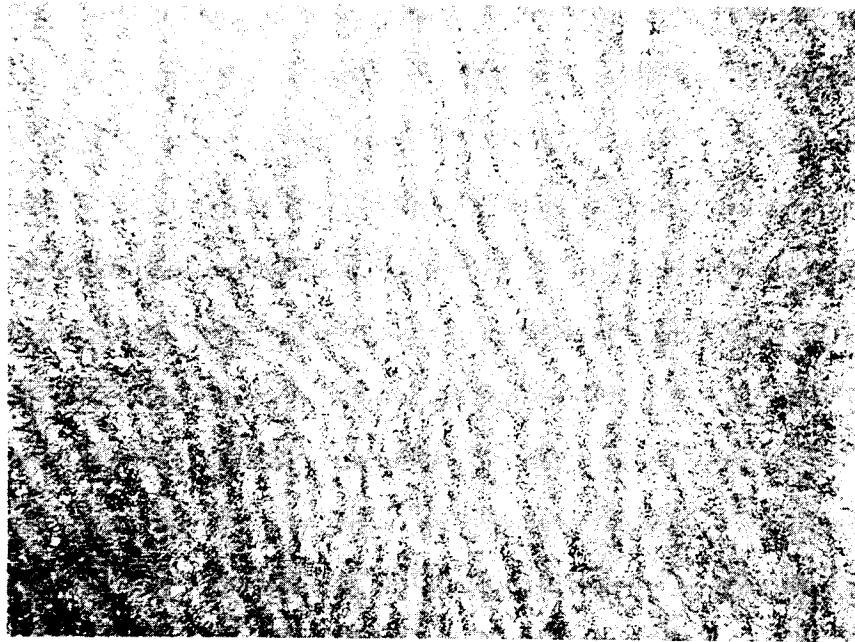
FIGURE III-2. Short Time Hot Tensile Strength of 0.100" Diameter Handy and Harman CuFo Wire (Reference: Handy and Harman Product Literature, 1964)

Tensile Strength - CuFo - (Copper - BeO)



**FIGURE III-3. 750°F Stress Versus Time to Rupture for Handy and Harman's CuFo Dispersion Strengthened Copper Compared to the 600° and 800°F properties of Standard Beryllium Copper No. 10 Alloy. (Reference: Handy and Harman Product Literature on CuFo (1964) and Westinghouse Data on Beryllium Copper, 1963)**

**Dispersion Strengthened Copper 0.375 inch As-received Material -  
2-5-64 Raw Stock For Orders 34789 & 34790**



**N-538**

**Transverse section**

**Mag. 100X**



**N-539**

**Longitudinal section**

**Mag. 100X**

**FIGURE III-4. Micrographs of 0.375 inch Diameter CuFo, BeO Dispersion Strengthened Copper**

## SECTION IV

### ELECTRICAL INSULATION MATERIALS

#### A. INTRODUCTION.

The gathering, determining, and interpreting of mechanical and physical property data on electrical insulating materials is moving into the testing phase. The materials are divided in two main classes; organic and inorganic. Wire insulation, flexible sheet, laminate, molded, and cast insulations are included in both classes. Much data have been collected for some of the applications. It should be pointed out that in many cases, although information is available, it has not been possible to determine the exact composition tested or the specific method used to obtain the data. In other cases, information may be available from several sources, but the data may be conflicting. In these problem areas, the technical knowledge of Westinghouse and others in the pertinent field will be drawn upon in an effort to interpret the data. Where necessary, new and controlled tests will be performed.

#### B. SUMMARY OF EFFORT IN THIS QUARTER.

During the second quarter of the program, compilation and interpretation of the collected data has continued. Based on the data study, candidate materials have been selected for each category. The tests for each category of insulation have been established and submitted in test book form to NASA. Tests under this program are in progress.

## C. TECHNICAL DISCUSSION.

The materials and pertinent data are discussed in the order of their tabular presentation.

### 1. Magnet Wire Organic Insulation

Magnet wire insulated with polyimide enamel (known as DuPont ML) is available from all major magnet wire producers. While some individual variations may be noted, all correspond in general to Military Standard Mil-W-583, Class 220, Type M2. The data which is being compiled at this time has been gathered from suppliers' correspondence and published data as well as Westinghouse tests. The data indicate that this insulation is mechanically strong and its thermal endurance is outstanding. Heavy enamel (double build) exhibits greater than 10,000 hour insulation life at 428°F. The most significant difference in polyimide insulated wire from various suppliers has been in weight-loss values. This property is dependent upon degree of resin cure and must be controlled. However, agreement on the most suitable level has not been reached, partly because of the variety of end applications for which the wires are intended. This and other variations will be discussed in a subsequent report.

### 2. Flexible Sheet-Organic

#### a. Polyimide Glass Sheet Insulation

Polyimide glass sheet insulation is produced by DuPont and is known as Pyre-ML. At the initiation of this program, one general grade was established, known as 6500, 6507, and 6508, depending upon sheet thickness. In this grade, the glass fibers are well wetted by the resin and the resulting structure is dense and electrically strong. This superior resin penetration and wetting gave rise to a problem in winding small, highly stressed configurations. This problem was severe fiber breakage and subsequent resin cracking, yielding low electrical strength values. New grades, identified as 6510, 6517, and 6518, were developed to overcome this problem in which the surface fibers are wetted but resin penetration is greatly reduced. Flexibility and retention of electrical strength after bending have been increased. The new grade of Pyre-ML is however not an improvement for applications operating at high frequency (megacycles), because of the multiplicity of voids within the sheet. Samples of both the 6508 and 6518 materials have been tested. Initial electrical strength data are reported in Table IV-1.

TABLE IV-1. Polyimide - Glass Sheet Insulation

Identification	Electrical Strength		Thickness	
	Test No.	Test No.	Test No.	Test No.
6508	1 - 6900 Volts	6 - 7300 Volts	1 - 0.0098 Inch	6 - 0.0102 Inch
	2 - 6800 Volts	7 - 7500 Volts	2 - 0.0098 Inch	7 - 0.0102 Inch
	3 - 6300 Volts	8 - 7300 Volts	3 - 0.0101 Inch	8 - 0.0102 Inch
	4 - 7200 Volts	9 - 6700 Volts	4 - 0.0102 Inch	9 - 0.0096 Inch
	5 - 6800 Volts	10 - 6000 Volts	5 - 0.0103 Inch	10 - 0.0095 Inch
	Average 6890 Volts Average Volts per mil - 690		Average 0.00999 Inches	
6518	1 - 4800 Volts	6 - 5300 Volts	1 - 0.0091 Inch	6 - 0.0101 Inch
	2 - 5600 Volts	7 - 6100 Volts	2 - 0.0100 Inch	7 - 0.0101 Inch
	3 - 5200 Volts	8 - 5600 Volts	3 - 0.0103 Inch	8 - 0.0101 Inch
	4 - 6200 Volts	9 - 5000 Volts	4 - 0.0097 Inch	9 - 0.0100 Inch
	5 - 5800 Volts	10 - 5500 Volts	5 - 0.0101 Inch	10 - 0.0098 Inch
	Average 5510 Volts Average Volts per mil - 551		Average 0.00998 Inches	
<b>Procedure:</b> The electrical strength of samples was measured using two inch electrodes and a Westinghouse tester of 8000-16000 volts capacity. The average thickness of the samples were found by measuring ten thicknesses along the entire sample length with a micrometer. Rise of voltage was approximately 1000 volts per second. Measurements shown were made at room temperature.				

Reference: Westinghouse Internal Laboratory Report No. 6-64



b. Polyimide Film Insulation

Polyimide film insulation is known commercially as H-Film and is produced by DuPont. It displays electrical strength values of about 1000 volts per mil, good thermal, chemical, and nuclear radiation resistance. DuPont and Westinghouse data are being combined and required tests are being initiated at this time to fill whatever information gaps exist.

3. Rigid Sheet, Laminate, Organic

a. Imide-Glass Laminate

Imide-glass laminate, known as I-8, was discussed in the first quarterly report. Material for the additional required tests is being prepared at this time.

b. Polybenzimidazole-Bonded Glass Laminate

The polybenzimidazole-bonded glass laminate called Imidite and produced by Narmco has been tested previously by Westinghouse. The tests were performed on Imidite 1850 and are reported in Table IV-2.

c. Epoxy-Glass Laminate

The epoxy-glass laminate, Westinghouse grade H2497, exhibits the properties reported in Table IV-3.

d. Diphenyl Oxide-Glass Laminate

Diphenyl oxide-glass laminate, Westinghouse grade H17511, containing grade 181 glass fabric exhibiting properties as collected from reference RI218, is reported in Table IV-4.

e. Phenolic Laminate

The phenolic laminate, 91 LD, developed by Cincinnati Testing Laboratory is partially described by the properties reported in Table IV-5.

TABLE IV-2. Polybenzimidazole Bonded Glass Laminate

Air Aging Time (hrs)	Aging Temp. (°F)	Test Temp. (°F)	Flexural Strengths (psi)		
			SAMPLE		
			1	2	3
0	--	70	108,350	114,250	--
1/2	600	600	116,400	115,100	--
24	600	600	80,350	94,150	64,900
100	600	600	23,300	26,400	21,400
250	600	600	3,350	2,550	2,500
250	600	600	3,100	3,500	3,700
298	600	600	1,200	1,150	1,200
298	600	70	1,450	1,450	700
<p>Imidite performs well for short times at elevated temperatures. However, between 24 hours and 100 hours, a distinct change occurs at 600°F, and the laminate rapidly disintegrates. For this reason, it is necessary to establish its maximum temperature limit somewhere below 600°F.</p>					

Reference: RI225

TABLE IV-3. Epoxy Glass Laminate

Test	Comments	Results	*Reference
Volume Resistivity		1.8 x 10 <sup>6</sup> megohm-cm	RI253
Electrical Strength	The specimens were conditioned prior to testing at 122°F for 48 hours. Perpendicular to laminate Parallel to laminate	60 KV 55 KV	RI253 RI254 RI253 RI254
Dielectric Constant	The specimens were conditioned prior to testing at 122°F for 48 hours. 1 megacycle	4.5	RI253
Power Factor	The specimens were conditioned prior to testing at 122°F for 48 hours. 1 megacycle	0.013	RI253
Impact	Lengthwise Crosswise	16.3 ft lbs/in 15.3 ft lbs/in	RI253
Water Absorption	This sample preconditioned 1 hour at 221°F This sample preconditioned 24 hours at 74°F See Figures IV-1, 2, 3, 4	0.08% 0.05%	RI253
Aged Flexural Strength	See Figures IV-5, 6, 7, 8		
Insulation Life	See Figures IV-9, 10, 11, 12		
*Refers to references at the end of this section.			

TABLE IV-4. Diphenyl Oxide Glass Laminate (Sheet 1 of 3)

<u>Volume Resistivity</u>		6.4 x 10 <sup>7</sup> megohm-cm
<u>Electric Strength at 74°F</u>	The specimens were conditioned for 48 hours at 74°F at 50% relative humidity prior to testing. Voltage stress was applied perpendicular to the laminations.	
<u>Method</u>	<u>Environment</u>	<u>Breakdown Voltage (Volts/Mil)</u>
Short time	Oil	408
Step by step	Oil	579
Short time	Air	416
Step by step	Air	667
<u>Dielectric Constant (1 mc)</u>	The first specimens were conditioned for 48 hours at 74°F at 50% relative humidity prior to testing. The second specimens were immersed in water at 74°F for 24 hours prior to test.	
	1.	4.94
	2.	5.00
<u>Power Factor (1 mc)</u>	The pre-test conditioning was the same as that for dielectric constant above.	
	1.	0.009
	2.	0.011
<u>Impact</u>	The specimens were conditioned at 122°F for 48 hours prior to testing.	
<u>Direction of Impact</u>	<u>Result (Ft-lb/in)</u>	
Edgewise, lengthwise	7.0	
Edgewise, crosswise	7.0	

TABLE IV-4. Diphenyl Oxide Glass Laminate (Sheet 2 of 3)

<u>Tensile Strength</u>		The specimens were conditioned for 48 hours at 74°F at 50% relative humidity prior to testing.
<u>Direction of Force</u>	<u>Result (psi)</u>	
Lengthwise	45,075	
Crosswise	42,295	
<u>Compressive Strength</u>		The specimens were conditioned for 48 hours at 74°F at 50% relative humidity prior to testing.
<u>Direction of Force</u>	<u>Result (psi)</u>	
Flatwise	70,726	
Edgewise, lengthwise	51,625	
<u>Flexural Strength</u>		The specimens were conditioned for 48 hours at 74°F at 50% relative humidity prior to testing.
<u>Direction of Force</u>	<u>Result (psi)</u>	
Flatwise, lengthwise	72,200	
Flatwise, crosswise	70,450	
<u>Water Absorption</u>		This specimen was conditioned for one hour at 221°F.
		<u>Result (psi)</u>
		0.132%
<u>Density (as specific gravity)</u>		This specimen was conditioned for 24 hours at 74°F and 50% relative humidity.
		<u>Result</u>
		1.81

TABLE I V-4. Diphenyl Oxide Glass Laminate (Sheet 3 of 3)

<u>Arc Resistance</u>	Same conditioning as density.	<u>Result</u>
		185 sec.
<u>Aged Flexural Strength</u>	See Figure I V-13, 14	
<u>Insulation Life</u>	See Figure I V-15	

Reference - RI218

TABLE IV-5. Phenolic Glass Laminate (91LD) (Sheet 1 of 2)

				Reference *
<u>Dielectric Constant</u>	<u>T(°F)</u>	<u>Frequency(mc)</u>	<u>Dielectric Constant</u>	
	77	1	4.0	RI509
	77	10	3.57	
after 1/2 hr at 500°F	500	1	3.98	
	500	10	4.16	
after 200 hr at 500°F	500	1	2.60	
<u>Power Factor</u>	<u>T(°F)</u>	<u>Frequency(mc)</u>	<u>sin δ</u>	
	77	1	0.0098	RI509
	77	10	0.010	
after 1/2 hr at 500°F	500	1	0.0055	
	500	10	0.0126	
after 200 hr at 500°F	500	1	0.0048	
<u>Impact-Izod</u>	15 ft-lb/in at 500°F after 1/2 hr at 500°F			RI517
	15 ft-lbs/in at room temp			
<u>Tensile Strength</u>	45,860 psi See Fig. I V-16 and 17 for heat aging data			RI516
<u>Flexural Strength</u>	67,500 psi See Fig. I V-18 for heat aging data			
<u>Compressive Strength</u>	65,800 psi See Fig. I V-19 for heat aging data			RI516
<u>Density (as Sp. Gravity)</u>	1.95			
<u>Thermal Conductivity</u>	0.15 BTU-ft/hr- °F-ft <sup>2</sup>			RI509
<u>Thermal Expansion</u>	5.5 x 10 <sup>-6</sup> in/in- °F			RI509

TABLE IV-5. Phenolic Glass Laminate (91LD) (Sheet 2 of 2)

		<u>Reference *</u>
<u>Flexural Modulus</u>	3.78 (10) <sup>6</sup> psi (Room Temperature) See Fig. IV-20 for heat aging data	RI516
<u>Weight Loss vs Temperature</u>	See Fig. IV-21	RI516
<u>Aged Flexural Strength</u>	See Fig. IV-17, 18	RI509 RI516

\* Refers to references at the end of this section.



4. Molding Compounds - Organic

a. Polyimide Molding Compound (SP Resin)

Considerable data have been gathered on the polyimide molding compound known as SP Resin and produced by DuPont. These data are reported in Table IV-6. Highly complex shapes are not molded satisfactorily using SP, but machining can be easily performed, and this material can therefore be considered for critical molded insulation requirements.

b. Epoxy Premix

Properties of epoxy premix, Scotchply 1100, are reported in Table IV-7.

c. Polyester Premix

The properties collected to date of the selected polyester premix, Plaskon 452, are reported in Table IV-8.

5. Encapsulating Compounds - Organic

a. Epoxy

The property values of interest for the epoxy casting compound made by Hysol and identified as C9-4186 resin and H5-3537 hardener are reported in Table IV-9. Values from several sources for volume resistivity, power factor, and dielectric constant do not agree. These properties will be determined as a part of this program.

6. Magnet Wire - Inorganic

Samples of the following subject wires have been acquired and some testing is in progress. The wires and producers are identified as:

- a. Anaconda - Anacote
- b. Anaconda - Anadur
- c. Phelps-Dodge - Ceramiceze
- d. Westinghouse - R2554B

TABLE IV -6. Polyimide Molding Compound (SP) (Sheet 1 of 4)

		Reference*
<u>Volume Resistivity</u>	1 to 2 x 10 <sup>16</sup> ohm-cm	RI120
	1.6 x 10 <sup>15</sup> ohm-cm	RI121
<u>Electric Strength</u>	80 mils 570 Volts/mil	RI121
	3 mils 4100 Volts/mil	RI121
<u>Dielectric Constant</u>	Frequency at 77°F at 212°F at 392°F	RI123
	60 cps 3.26 3.24 3.18	
	1000 cps 3.24 3.24 3.16	
	T(°F) Freq. (mc) Dielectric Constant	RI123
	74 1 3.7	
	212 1 3.6	
	347 1 3.4	
	437 1 3.4	
	527 1 3.4	
	572 1 3.4	
	T(°F) Freq. (cps) Dielectric Constant	
	74 100 3.47	
	74 10,000 3.46	
NOTES:	1. At 10 <sup>6</sup> cps-dried at 302°F for 2 hours then tested over range of 74°F to 572°F yielded a dielectric constant value of 3.4.	
	2. At 10 <sup>6</sup> cps-with 3.2% absorbed water then tested at 74°F yielded a dielectric constant value of 4.82.	

TABLE IV-6. Polyimide Molding Compound (SP) (Sheet 2 of 4)

	Reference*			
Power Factor	Frequency	77°F	sin $\delta$ 212°F	392°F
	60	0.0013	0.0005	0.04
	1000	0.0016	0.0005	0.003
	Temp. (°F)	Freq. (mc)	sin $\delta$	
	74	1	0.008	
	212	1	0.007	
	347	1	0.003	
	437	1	0.003	
	527	1	0.008	
	572	1	0.05	
	T(°F)	Freq. (cps)	sin $\delta$	
	74	100	0.0015	
Impact Strength (Izod)	74	10,000	0.0022	
	At 10 <sup>6</sup> cps with 3.2% absorbed water sin $\delta$ = 0.039			
	0.7 to 1.1 ft-lb/in (room temp.)			
Tensile Strength	0.55 ft-lb/in (room temp.)			
	T(°F)	Tensile (psi)	Elongation (%)	
	77	13,000 $\pm$ 2000	4.5	
	302	9700	4.8	
	482	7700	4.6	
	600	5000	-	
	752	3500	-	
Flexural Strength	T(F°)	(psi)		
	73	14,700		
	509	8,000		

TABLE IV-6. Polyimide Molding Compound (SP) (Sheet 3 of 4)

			Reference*
<u>Compressive Strength (RT)</u>	24, 400 psi		RI121
<u>Flexural Modulus</u>	See Fig. IV-22		RI121
<u>Water Absorption</u>	in 50% Relative Humidity 2.1% in boiling water 3.2%		RI121
<u>Density (as specific gravity)</u>	1.4 to 1.43		RI121
<u>Thermal Conductivity</u>	0.184 BTU-Ft/Hr-°F-Ft <sup>2</sup>		RI121
<u>Thermal Expansion (annealed)</u>	2.84 x 10 <sup>-5</sup> in/in-°F (-300 to 500°F)		RI121
<u>Chemical Resistance</u>	organic solvents-insoluble boiling water-thin film embrittles slightly strong acid-resistant strong bases-attached		RI120
<u>Arc Resistance</u>	185 seconds (tracks)		RI121
<u>Elastic Modulus</u>	430, 000 psi		RI121
<u>Abrasion Resistance</u>	Bearing properties PV Limit 110, 000 Wear constant 40 x 10 <sup>-10</sup> Wear against 1025 mild steel-excellent		RI120 RI120
	Coefficient friction (1025 steel, in air at RT velocity 834 ft/min) = 0.08 to 0.015		RI121

TABLE IV-6. Polyimide Molding Compound (SP) (Sheet 4 of 4)

<u>Abrasion Resistance</u> (Cont.)	Coefficient plane sliding friction for polished SS 1/2"/min and 1/16 psi is 0.17 at atmosphere pressure and 0.13 at $5 \times 10^{-8}$ mm Hg.	<u>Reference*</u>
<u>Fatigue Endurance Limit</u>	5800 psi (axial fatigue-peak alternating stress $10^6$ cycles)	RI120
<u>Radiation Resistance</u>	Threshold damage $7 \times 10^9$ rads (2 mev Van deGraf electrons)  1500 hours at 347°F for total $10^{11}$ rads embrittled	RI120  RI120
<u>Heat Distortion</u>	(264 psi) at 473°F	RI121
<u>Shrinkage</u>	Deformation under load (2000 psi) at 122°F = 0.5%	RI121
*Refers to references at the end of this section.		

TABLE IV-7. Epoxy Premix

(Scotchply 1100)

		<u>Reference*</u>
<u>Volume resistivity (500 Vdc)</u>	$3.8 \times 10^{15}$ ohm-cm	RI160
<u>Electric Strength</u>	360 Volts/mil	RI160
<u>Dielectric Constant</u>	5.5 (100 cps)	RI160
<u>Power Factor</u>	0.087 (100 cps)	RI160
<u>Impact (Izod)</u>	25 ft-lb/in	RI160
<u>Tensile Strength</u>	See Fig. I V-23	RI160
<u>Flexural Strength</u>	62,000 psi (See Fig. I V-25)	RI160
<u>Compressive Strength</u>	38,000 psi	RI160
<u>Water Absorption</u> (24 hour immersion)	0.053%	RI160
<u>Density (as specific gravity)</u>	1.8	RI160
<u>Thermal Conductivity</u>	0.208 BTU-Ft. /Hr-°F-Ft <sup>2</sup>	RI160
<u>Thermal Expansion</u>	$6.03 \times 10^{-6}$ in/in-°F	RI160
<u>Arc Resistivity</u>	125 sec	RI160
<u>Flexural Modulus</u>	See Fig. I V-24	RI160
<u>Shrinkage</u>	0.0005 in/in	RI160
*Refers to references at the end of this section.		

TABLE IV-8. Polyester Premix

(Plaskon 452)

<u>Electric Strength</u>	Short time Step by step	375 Volts/mil 325 Volts/mil
<u>Dielectric Constant</u>	60 cps	6.5
	1000 cps	6.1
	10 <sup>6</sup> cps	5.9
<u>Power Factor (sin δ )</u>	60 cps	0.015
	1000 cps	0.009
	10 <sup>6</sup> cps	0.011
<u>Impact (Izod)</u>		2.5 ft-lb/in
<u>Tensile Strength</u>		5700 psi
<u>Flexural Strength</u>		13,500 psi
<u>Compressive Strength</u>		30,500 psi
<u>Water Absorption 24 hrs @ 74°F</u>		0.08%
<u>Density (as Specific Gravity)</u>		2.23
<u>Thermal Conductivity</u>		0.244 BTU-Ft. /Hr- °F-Ft
<u>Thermal Expansion</u>		20 x 10 <sup>-6</sup> in/in- °F
<u>Arc Resistance</u>		>120 sec
<u>Shrinkage</u>		0.002 in/in
Note: This data extracted from Reference RI174*		
*Refers to references at the end of this section.		

TABLE IV-9. Epoxy Casting Compound  
(Hysol-C9-4186 resin, H5-3537 hardener)

<u>Electric Strength</u>	650 Volts/mil	<u>Reference*</u> RI240
<u>Tensile Strength</u>	8000 psi	RI240
<u>Flexural Strength</u>	12,000 psi	RI240
<u>Compressive Strength</u>	28,000 psi	RI240
<u>Water Absorption</u>	0.2%	RI240
<u>Density</u>	0.059 lb/in <sup>3</sup>	RI240
<u>Thermal Conductivity</u>	0.22 BTU-Ft. /Hr- °F-Ft <sup>2</sup>	RI240
<u>Thermal Expansion</u> (86 to 194°F)	56 x 10 <sup>-6</sup> in/in-°F	RI240
<u>Shrinkage</u>	0.007 in/in	RI240
*Refers to references at the end of this section.		



The data thus far collected is being evaluated at this time and is not ready for inclusion in this report.

Performance data will be reported on plasma-sprayed aluminum oxide coating on magnet wire. This available data is being gathered from the project "Alkali Metal Resistant Wire" contract AF33(657)10701. Additional samples of this type wire will be prepared and tested under this program to fill gaps in the data.

#### 7. Lead Wire - Inorganic

The lead wires selected for this program are as listed below:

- a. Asbestos braid over nickel plated copper from Continental Wire Corp.
- b. Micatemp - from Rockbestos Wire and Cable Co.
- c. Super-Super Jet from Boston Insulated Wire and Cable Co.

Performance data is being collected at this time for these specimens. Additional wire is being acquired.

#### 8. Flexible Sheet Insulation - Inorganic

Samples of the following materials have been acquired:

<u>Construction</u>	<u>Trade Name</u>	<u>Supplier</u>
a. Mica paper	Burnil	Minnesota Mining & Manufacturing Co.
b. Mica-glass-phosphate-bond	ABIPM	Westinghouse
c. Mica-glass-silicone-bond	128-50-1	Westinghouse
d. Silicate fiber paper	Fiberfrax	Carborundum

Long term aging tests required for this program are being initiated during March and April.

#### 9. Rigid Sheet and Molded Rigid Parts - Inorganic

The rigid sheet insulations selected for this program have either been already acquired or are on order. They are listed in Table IV-11. The compilation of data from the literature is not complete at this time.

This task has been very difficult because of wide variation in test results and methods. These variations rise from the many processing variables and trace impurities. In addition, much of the data is reported with insufficient sample composition and history. Tabular presentations of these conditions are being prepared and some correlation of the factors will be attempted.

10. Encapsulation Compounds - Inorganic

The four compounds named in this group in Table IV-11 have been acquired. Preparation of specimens is in progress.

#### **D. FINAL MATERIAL SELECTION .**

The materials and grades selected for this program are listed in Tables IV-10, Organic Insulations, and IV-11, Inorganic Insulations. The selections are based upon comparative study of trade and suppliers data, and Westinghouse data. The selections represent judgement of the collective effect of such factors as material performance, availability, and intended applications.

A comparison of Table IV-10 and IV-11 with Table II-4 and II-5 in the previous Quarterly Report (WAED 63. 16E) provides an indication of the data acquired during this quarter from the literature and from testing.

TABLE IV-10. Organic Insulations

Property	Insulated Wire		Flexible Sheet				Rigid Sheet - Laminate				Molded Rigid Parts				Encapsulation Compounds			
			Polyimide		Polyimide Film		Polyimide		Polyimide		Imide		Epoxy		Polyester		Epoxy	
	RT	MT	RT	MT	RT	MT	RT	MT	RT	MT	RT	MT	RT	MT	RT	MT	RT	MT
Volume Resistivity DC	X*																	
60 cps																		
400 cps																		
1600 cps																		
3200 cps																		
Electric Strength DC																		
60 cps	X	X	X	X														
400 cps																		
1600 cps																		
3200 cps																		
Dielectric Constant																		
60 cps	(10 <sup>2</sup> )	(10 <sup>2</sup> )	X															
400 cps	X	X																
1600 cps	(10 <sup>3</sup> )	(10 <sup>3</sup> )																
3200 cps	(10 <sup>5</sup> )	(10 <sup>5</sup> )																
Power Factor	*	*																
400 cps																		
1600 cps																		
3200 cps																		
Thermal Shock	X	X	*															
Impact Strength	NA	NA	NA	NA	NA	NA												
Tensile Strength	NA	NA	X	X	X	X	T	T										
Rupture Mod. or																		
Flexural Strength	NA	NA	NA	NA	NA	NA	X	X	X	X	X	X	X	X	X	X	X	X
Compressive Strength	NA	NA	NA	NA	NA	NA	T	T	T	T								
Porosity (Water Abs.)	NA	NA	NA	NA	NA	NA												
Density	NA	NA	NA	NA	NA	NA												
Thermal Conductivity	*	*	*	*	*	*	T	T										
Thermal Expansion	*	*	*	*	*	*	T	T										
Chemical Resistance	X	X	X	X	X	X												
Arc Resistance	NA	NA	NA	NA	NA	NA												
Elastic Mod. (Flex.)	NA	NA	NA	NA	NA	NA	T	T										
Abrasion Resistance	X	X	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Cut-thru Resistance	X	X	X*	X*	X*	X*	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sublimation Rate																		
(Wt. Loss)																		
Radiation Resistance																		
Endurance (Post-aging Flex.)	NA	NA	NA	NA	NA	NA												
Insulation Life	X	X																
Adhesion	X	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Shrinkage	NA	NA	NA	NA	NA	NA												
Compatibility																		
Manufacturer	Major Wire Suppliers		DuPont				Westing-house				Westing-house							
Trade Name	Various	Pyre-M/L ***	H-Film				I-8				Narmco Imidite ***							
											H2497							
											Doryl H17511							
											CTL**							
											Dupont SP							
											3M							
											Scotchply 1100							
											Allied Plaskon 452							
											Hysol C9-4186							
											Carbin H5-3537							
											Carbin XRS017							
											3M							

NOTES: X = Data Available RT = Room Temperature MT = Maximum Use Temperature T = Tests Now in Progress  
 ( ) = Another Frequency Available \*\*\* = Cincinnati Testing Laboratory \*\*\* = See Discussion, Sect. IV C3

TABLE IV-11. Inorganic Insulations

Property	Insulated Magnet Wire		Insulated Flexible Wire (Stranded)		Flexible Sheet										Rigid Sheet										Molded Rigid Parts						Encapsulation Compounds																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																						
	Insulated Magnet Wire		Insulated Flexible Wire (Stranded)		Synthetic Mica Paper		Mica-Glass Phosphate Bond		Mica-Glass Silicone Bond		Silicate Fiber Paper		Mica Laminates		Asbestos BPO4		Alumina 0.25MgO		Beryllia 96%		Mica-Glass Bonded		Alumina 94%		Alumina 99%		Beryllia 99.8%		Mica-Glass Bonded		Refractory Oxides With Inorganic Bonding Materials																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																						
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NOTES: X = Data Available P = Partial Data Available NA = Not Applicable RT = Room Temperature HT = High Temperature T = Tests in Progress \* = Non-Standard Test  
( ) = Another Frequency Available MC = Megacycle KMC = Kilomegacycle

TABLE VI-1. Gas Transfer (Mol %)

Temperature	H <sub>2</sub>	CO	N <sub>2</sub>	O <sub>2</sub>	CO <sub>2</sub>	Other Hydrocarbons
Room Temp.	6.63	--	30.39	8.23	31.35	23.40
1600°F	34.19	22.92	14.79	3.41	10.19	14.5

diffusion pump. Typical leak rates at room temperature and 1600°F were  $1 \times 10^{-6}$  cc-atm/sec. For some tests, the leak rate observed was higher at 1600°F than at room temperature and in other tests, the leak rate was lower. The validity of such leak rate measurements can be questioned unless it is established that the pumping speed of the system is zero. This is not usually the case, since pumping with condensed oil films deposited within the test chamber from distilled oil is inherent with an oil diffusion pumped system, and chemical pumping of certain gases by metals at 1600°F is inherently a part of oxide, nitride, and carbide formation. The lower leak rate values obtained with the ionization gage as compared to the cryogenic pump collection suggests that the diffusion pump is successfully competing with other pumping mechanisms in the system in transferring a fraction of the total gas from the system.

The theoretical maximum pumping speed for any pump has been calculated from kinetic theory and is 12.6 liters/sec per  $\text{cm}^2$  for air at room temperature. The large chamber area available at 1600°F for pumping at this possible rate would suggest that the system does not approach zero pumping speed as required for a true measurement using the rate of rise of pressure as a true leak measurement. Such a chemical pump is as much a pump (perhaps a better pump) for certain gases than is an oil diffusion pump for which the maximum efficiency is only 30% or about 4 liters/sec per  $\text{cm}^2$  of orifice area.

It is believed that the observation of gas transfer rates found by a cryogenic pump at the exhaust of the diffusion pump provides a better sampling of the system and provides a more direct measure of gas transfer rates to which a test specimen may be subjected than is the use of a pressure rate of rise measurement. The difference in leak rates between the two methods can differ by one to two orders of magnitude as observed in the tests just discussed.

## SECTION VII

### MECHANICAL AND ELECTRICAL DESIGN REQUIREMENTS FOR MATERIALS

#### A. INTRODUCTION.

The electrical components which make up an advanced space electric power system include transformers, motors, generators, contactors, and various control elements. A material guideline for the selection of magnetic, electrical conductor, electrical insulation, and bore seal materials for these components was presented in the First Quarterly Report (WAED63. 16E). These guidelines, which are design oriented, have provided the basis for specifying the material properties needed to successfully build an advanced space electric power system and to further define the importance which should be placed on the various properties. The design analysis has also served as a basis for recommending the methods which should be used in the data presentations.

This report will continue the analysis reported previously. The following sections present a simple bore seal mechanical analysis and an inquiry into the material needs of an exciter-regulator, a power transformer and a magnetic amplifier.



## B. BORE SEALS.

The initial efforts have been associated with the bore seal for the radial-gap machine. The determination of stresses, particularly in the ceramic-metal joints, requires the construction of mathematical models of the seal components. The first models have been constructed assuming no deflection in the quarter-toroidal sections between the cylindrical and diaphragm sections. A similar assumption was made in analysis of bellows sections.

The deflection of the resulting diaphragm section is illustrated in Figure VII-1.

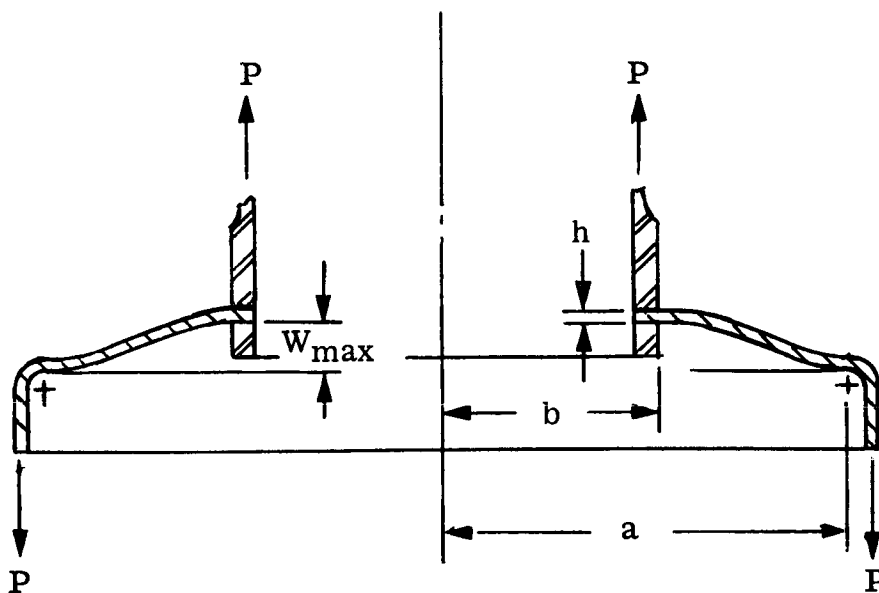


FIGURE VII-1. Bore Seal Stress Model

A solution for this type of loading and deflection provides the relationship:

$$W_{\max} = kPa^2/Eh^3 \quad (1)$$

where:  $k = f(a/b)$

$E$  = Modulus of elasticity and

$W_{\max}$  = total deflection of diaphragm

$P$  = Load.

The factor  $k$  increases as a function of the outside to inside radius ratio,  $a/b$ . The inner radius,  $b$ , and the product,  $Eh^3W_{\max}$ , may be considered constant for a given design. Then the axial load,  $P$ , will be inversely proportional to the product,  $ka^2$ , and can be significantly reduced by increasing the radial depth ( $a-b$ ) of the diaphragm.

When the diaphragm depth is limited by the generator design, a bellows can be used. This can be considered as a number of diaphragms (two per convolution) in series. The axial deflection per diaphragm will be  $W_{\max}/2n$  (where  $n$  is the number of convolutions and  $W_{\max}$  is the total deflection absorbed by the bellows).

The simplified analysis of diaphragms was applied to the welded bellows of Figure VII-2. A similar bellows had been tested under compressive loads, but assembled (welded) dimensions are unavailable. Drawing dimensions of the parts, with an assumed weld depth of 1/16 inch, were used in the analysis. Results are indicated with the test values in the table below. Three of the four calculated loads are within 6% of the test loads. While these calculations do not establish the accuracy of this approach, they do tend to support the suitability of the analysis for preliminary design estimates.

<u>Deflection (inch)</u>	<u>Test Load (lbs)</u>	<u>Calculated Load (lbs)</u>	<u>Deviation %</u>
0.0015	25	30.9	23.6
0.0016	35	33.0	-5.7
0.0022	45	45.4	0.9
0.0025	50	51.5	3.0

(1) S. Timoskenko, Strength of Materials, Part II, 3rd Edition, pp. 113-114, D. VanNostrand Co., Inc., 1956.

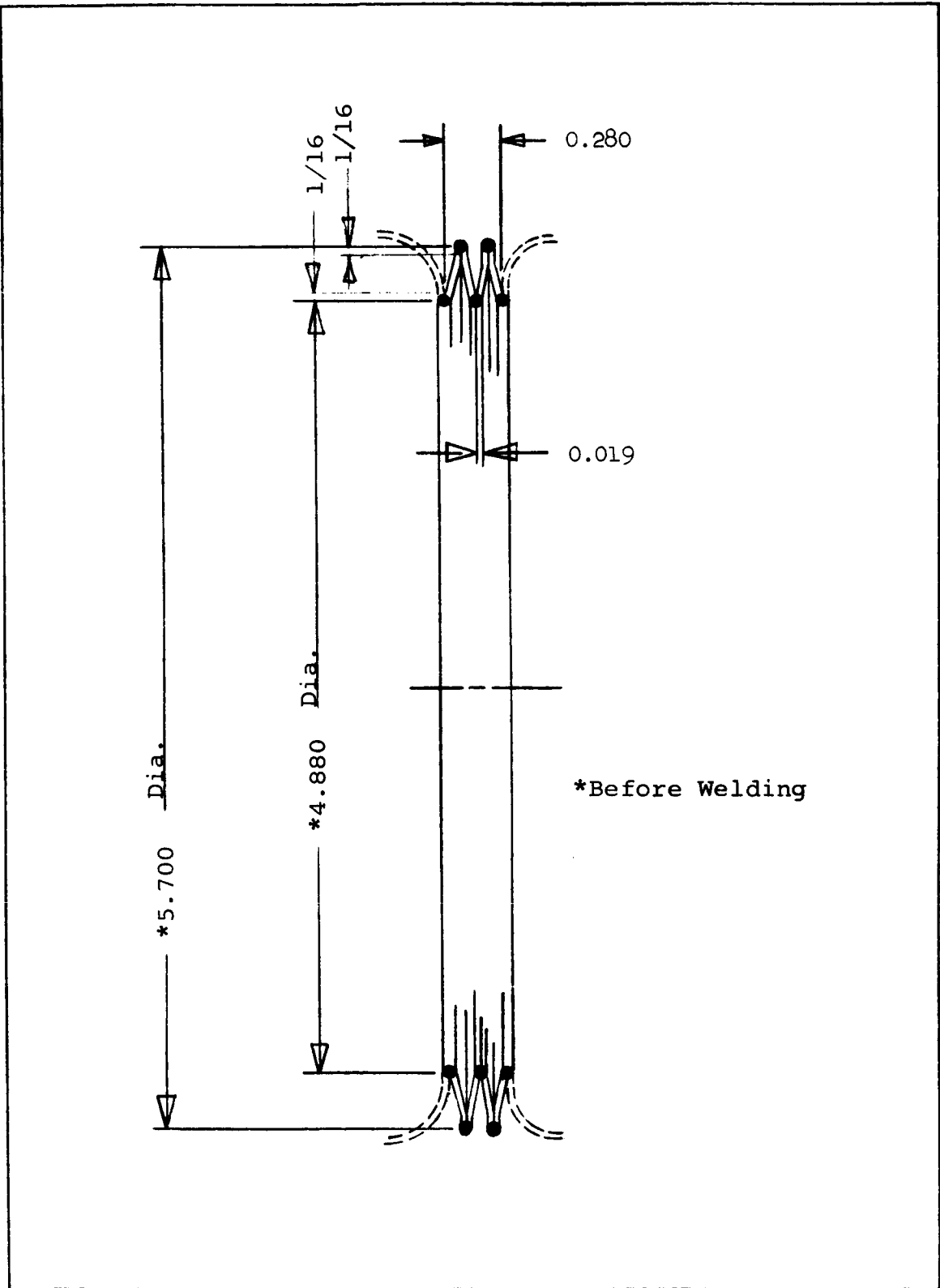


FIGURE VII-2. Welded Bellows

WAED64. 14E-152

The stress of the diaphragm is determined through the equation<sup>2</sup>,  $s = k_s P / h^2 = (E h k_s W_{\max}) / (k a^2)$ . The factor,  $k_s$ , also increases with the radius ratio,  $K_s/k$ , decreases with increasing radius ratio. Following the previous assumptions, the diaphragm stress, proportional to  $k_s / k a^2$ , will decrease with increasing diaphragm depth. The approximate stresses of the bellows diaphragms can be similarly determined.

<sup>2</sup>ibid. S. Timoskenko. Strength of Materials, Part II, 3rd Edition, pp. 113-114, D. VanNostrand Co., Inc., 1956.

### C. THE EXCITER-REGULATOR.

The exciter-regulator provides regulation and control for the electrical output of the a-c generator. The inductor a-c generator was selected<sup>3</sup> as the most probable type to operate in the environments experienced by advanced space electric power systems. The generator is necessarily close to the prime mover (a location which may be unsuitable for some system components) and will be designed to use liquid coolant at a temperature in excess of 450°F (but probably less than 1200°F).

The exciter-regulator must deliver approximately 10 KW of excitation power for a 1 megawatt system. A review of previous studies indicates that the best means of system regulation and control is the type of exciter-regulator described in Contract NAS5-1234<sup>4</sup>.

The exciter-regulator for this material definition study is the same except that the load division portion is omitted because load division is not required.

The usual exciter-regulator contains a power transformer to provide power for exciting the a-c generator field. The power transformer occupies a substantial portion of the exciter-regulator package and also contributes significantly to the losses. Further, available materials would permit building a transformer which can operate in the same environment as the generator and which can also use a coolant of 450 to 850°F. The power transformer has, therefore, been removed from the exciter-regulator package.

The small sensing and preamplifier transformers were not removed because they are small and have low losses. It is also desirable that they be close to the other elements of the exciter-regulator to eliminate the effect of false signals.

Figure VII-3 is a drawing of a typical exciter-regulator which can be used for a space electric power system. Connections are not shown but all major parts are included. These are listed below under their appropriate function.

#### 1. Error Detector

- Sensing Transformer
- \*Rectifier diodes
- \*Reference (Zener) diodes
- \*Resistors, variable and fixed

<sup>3</sup>Section IV A, Report No. WAED 63. 16E, First Quarterly Report, November 30, 1963, Contract No. NAS3-4162.

<sup>4</sup>Space Electric Power Systems Study, Final Report, Vol. 4, Contract No. NAS5-1234.

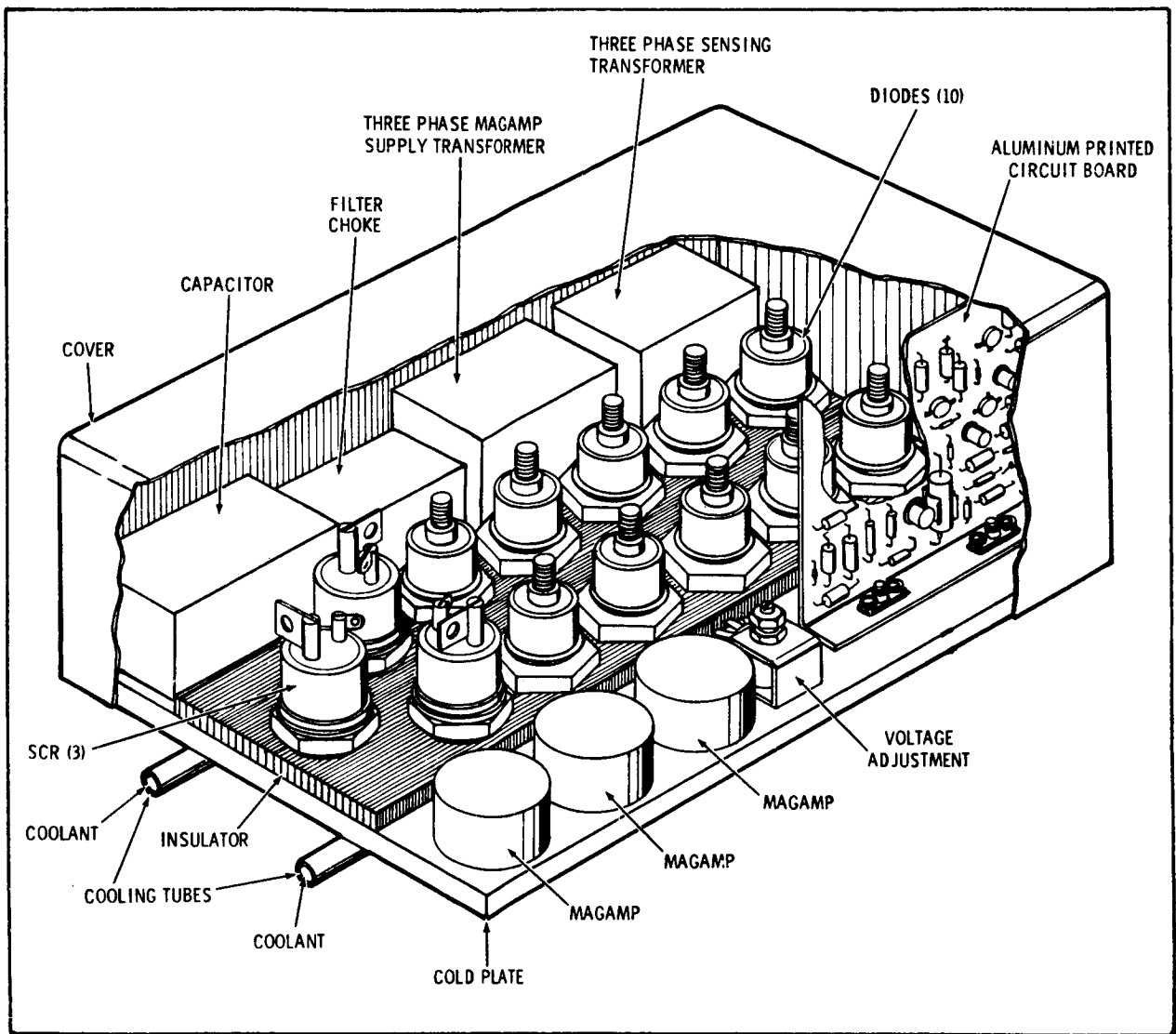


FIGURE VII-3. Typical Exciter-Regulator

## 2. Preamplifier

- Transformer
- Magnetic Amplifiers (3)
- \*Rectifier diode
- \*Zener diode
- Capacitor
- Choke
- Resistor for voltage adjustment

## 3. Power Amplifier

- Transformer (external)
- Rectifier diodes, 4 required
- Silicon controlled rectifier (SCR), 3 required

## 4. Auxiliary Rectifier

- Rectifier diodes, 6 required
- Current transformers (external)

\*Mounted on aluminum printed circuit board.

In the present state of the art, the rectifiers and diodes of the exciter-regulator are essentially low-temperature devices. Because of their intimate relation to other parts, the entire exciter-regulator becomes a low-temperature device which requires a coolant temperature of less than 120°F.

Without the power transformer, the exciter-regulator loss will not exceed 125 watts, and the volume is reduced to about 1/3 cubic foot. It is now possible to place it in any location where coolant can be supplied at the required temperature. If the location is where the parts may be damaged by coolant vapors or other gases, a sealed cover would be used and the interior filled with an inert gas.

## 1. Exciter-Regulator Cooling System

All parts of the exciter-regulator are mounted in such a way that the heat will be readily dissipated. Power diodes and silicon-controlled rectifiers are mounted on an insulating base which is also a good heat conductor, BeO, for example. This sheet insulation is in intimate contact with the cold plate to which cooling tubes are brazed. Internally-insulated parts are mounted directly on the cold plate. Small parts are mounted on the aluminum printed circuit board. Figure VII-4 shows the method of insulating and cooling the large silicon controlled rectifiers and diodes.

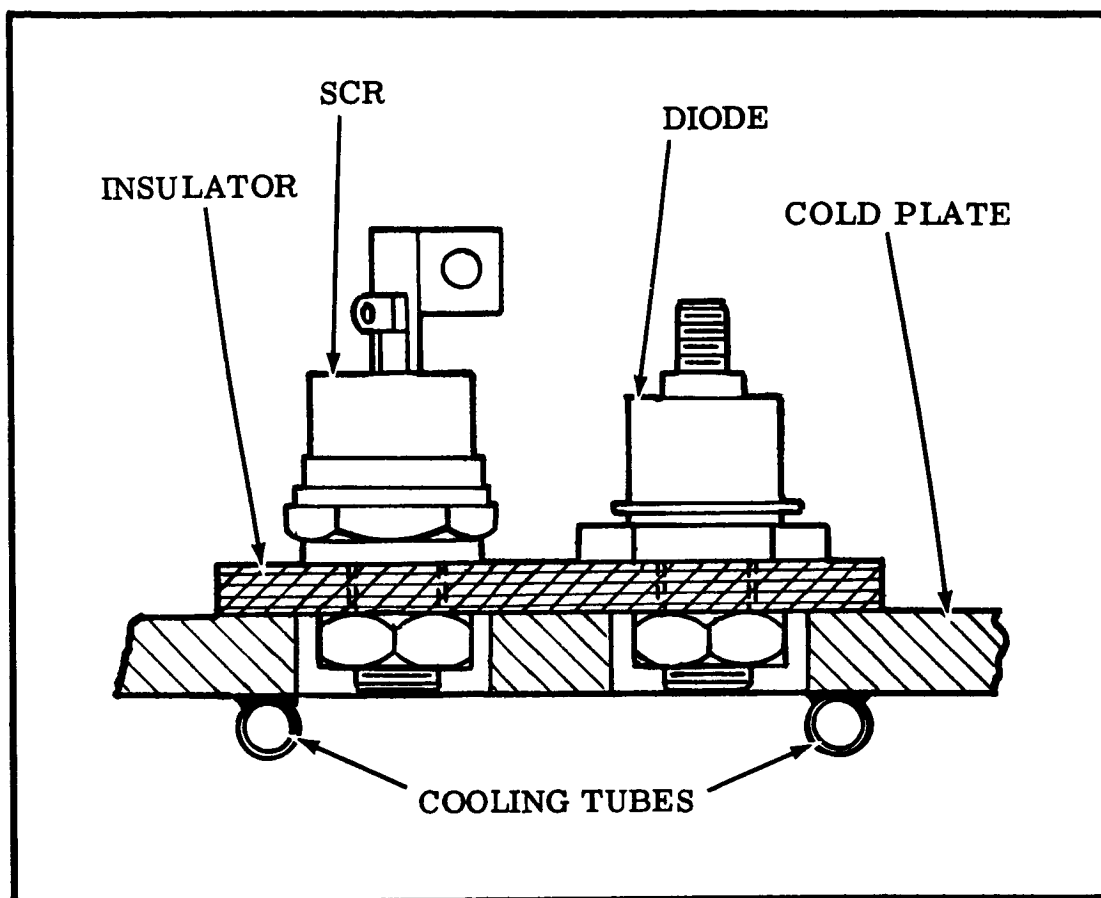


FIGURE VII-4. Insulation And Cooling of Silicon Controlled Rectifier And Diode



## 2. High Temperature Exciter-Regulator

There may be conditions in space electric power systems where the exciter-regulator is required to operate using a coolant temperature of 450 to 850°F, possibly higher. Development programs on silicon-carbide and gas-filled rectifiers are now underway. A 900°F, 10-ampere, silicon-carbide rectifier appears feasible based upon recent laboratory tests. The development of a high-temperature, controlled rectifier will be necessary to build a high-temperature exciter-regulator. Other parts of the exciter-regulator such as transformers, saturable reactors, capacitors, chokes, and resistors must be proven for high temperature operation. The present contract will define the performance for transformers, chokes, and saturable reactors. High-temperature exciter-regulators can become a reality after the components have been proven but this may be a penalty in the form of greater weight and volume.

## D. THE POWER TRANSFORMER.

In considering a space electric power system, the power transformer was removed from the exciter-regulator package to permit the building of a transformer which can operate in the same environment as the generator and which can also use a coolant of 450 to 1200°F.

The power transformer is a static device consisting of two or more coils of wire, a magnetic core, insulation, a cooling system, and means of holding the parts in place.

### 1. Transformer Coils

The coils of the transformer are wound with either magnet wire or foil, the selection depending on environment, cooling media, rating, and design considerations. A clad material is used to meet the system temperature requirements.

The derived characteristics of the conductors have been stated in the First Quarterly Report (WAED 63. 16E) on page 75.

### 2. Magnetic Core Material

The transformer core may be assembled either of tape or of punched laminations. Desired characteristics of the magnetic material are stated in the First Quarterly Report (WAED 63. 16E) on page 73. Low losses and exciting volt amperes per pound are very important in transformers; therefore, special consideration must be given to these properties in the material.

### 3. Insulation System

The transformer uses insulation in the following manner:

- a. Conductor insulation between adjacent turns
- b. Sheet insulation between layer and coils
- c. Sheet or insulation forms between coils and core
- d. Impregnants may be used to add rigidity and protection to the coil structure
- e. Potting material may be used for mechanical strength and to aid heat transfer.

At the operating temperature of a space electric power system and using a coolant in the range of 450 to 1200°F all insulation must be inorganic.

4. Coolant

The transformer will require a coolant, possibly a liquid metal in the range of 450 to 1200°F so that the temperature of all parts of the transformer will be within the limits of the materials used.

## E. THE MAGNETIC-AMPLIFIER.

The magnetic-amplifier is an essential part of the exciter-regulator for a space electric power system. It is a control device consisting basically of a tape-wound toroidal magnetic core, a control winding, an output or gate winding, and an insulation system. Additional windings may be used if the control system requires them.

Presently, magnetic-amplifiers are low-temperature devices because they include silicon rectifiers, limited to about 200°F. Recent research and development programs are focused on providing high-temperature rectifiers which can function with a high-temperature magnetic-amplifier.

The high-temperature magnetic-amplifier uses a square-loop, magnetic material, wound as tape, into a toroidal core. Magnetic materials are limited for this application, but a few show promise of being suitable for temperatures up to 1100°F.

The core is encased in an inorganic core which also serves as ground insulation for the windings.

The gate winding is wound on the insulating core box using a high-temperature wire having inorganic insulation between turns. The control winding is similar except that it is wound of smaller wire. Instead of being wound one on top of the other as in the transformer, the windings may be wound side by side in the core to simplify winding and insulation.

## SECTION VIII

### PROGRAM

#### A. OVERALL PROGRAM PLAN.

This program represents a 12 month effort ending the latter part of August, 1964, with additional reporting and publication time. The first major milestone of the program was reached with the publication of the previous quarterly report where the comprehensive literature search was reported. The present quarterly report has presented the test program which has been initiated to fill in the gaps observed from the literature evaluation. Figure VIII-1 presents the program as it is planned. The Westinghouse Aerospace Electrical Division is acting as the prime contractor and is being assisted by the Westinghouse Research and Development Center. The Eitel McCullough Corporation is a subcontractor and is supporting the bore seal analysis.

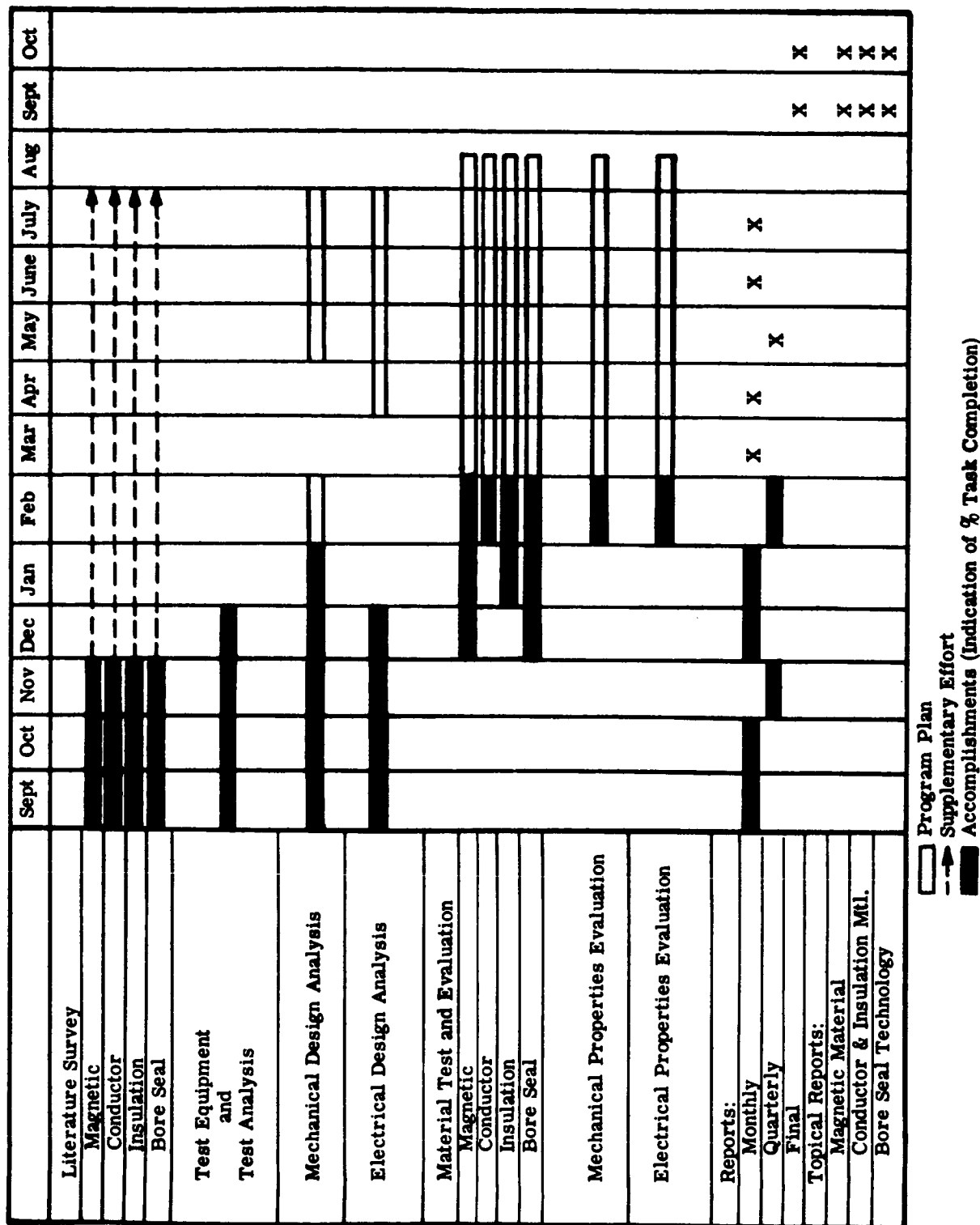


FIGURE VIII-1. Program Plan - NASA Materials Study and Research Program

## B. THIRD QUARTER PLAN.

### 1. Magnetic Materials

The creep and fatigue testing which was stated in the second quarter will be expanded considerably during the third quarter. Other mechanical and physical tests will be completed as specimens become available. It should be noted that specimens for long-time tests were given machining priority over such one-time, short-duration test specimens as specific heat and resistivity specimens.

Winding of the magnetic test samples for the high-temperature tests will begin in March.

### 2. Conductor Materials

Present plans call for the initiation of aging tests during April. The other thermophysical measurements outlined in the first quarterly report will be initiated concurrent with the aging tests.

### 3. Insulation Materials

During the third quarter of this program, several concurrent efforts will be in progress as follows:

- a. The plotting and tabulation of data will be continued.
- b. The acquisition and preparation of test materials will continue.
- c. Sublimation rate determinations will be started.
- d. Tests will be initiated as materials become available.

### 4. Bore Seal Materials

- a. Items which were not completed as forecast in the last quarterly report are repeated here with comments.
  - 1) Correlation of the modulus-of-rupture and tab-peel tests with ASTM CLM 15 tensile and peel tests. All parts have not yet been received due to a delay in supplier delivery. To minimize variables, all parts must be processed simultaneously. When the overdue parts are received, this geometry correlation study will be carried out.

- 2) Work will be initiated on the fabrication of the thermodynamically promising ceramic bodies into test pieces. Oxides of yttrium and dysprosium of 99.9% purity and less than 0.01% silica are on order but have not yet been received.
- b. The next quarter will see the loading of sample capsules for the ceramic screening test of 500 hours in K at 1600°F. The environmental test will be initiated. Purity test samples will be submitted for determination of loaded capsule alkali metal composition.
  - c. Preliminary mechanical and metallurgical screening of the following sealing systems will continue.
    - 1) Tungsten metalizing paints with alkali metal compatible non-metallic phase and nickel alloy braze to columbium alloy.
    - 2) Thin film metalizing - nickel alloy braze.
    - 3) Electroformed seals.
  - d. Active alloy brazing system development will be initiated on the high purity Group III beryllia body.



## APPENDIX A

### LEAK TEST CALIBRATION AND PROCEDURE

#### 1. Calibration:

A certified leak obtained from the ALO Standards Laboratory at Sandia, New Mexico is utilized to calibrate a secondary standard. The secondary standard is then used to check the sensitivity of the leak detectors daily. The specific calibration routine varies somewhat with the type and model of leak detector. Normal sensitivities range from 1 to  $3 \times 10^{-10}$  standard cc/sec and remain quite stable over a period of several weeks.

#### 2. Procedure:

- a. Use a leak detector which is checked daily for sensitivity. Sensitivity must be better than  $5 \times 10^{-10}$  standard cc/sec as determined with certified leak.
- b. Place sample on manifold connection and evacuate.
- c. Place bag or inverted jar to completely cover sample and manifold connection.
- d. Fill bag or jar with helium to flush out all air; continue helium flow through next step.
- e. Leak detector output meter must indicate no leak on the most sensitive scale.

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<p>Union Carbide Nuclear Company P. O. Box X Oak Ridge, Tennessee Attn: X-10 Laboratory Records Dept.</p> <p>United Nuclear Corporation Five New Street White Plains, New York Attn: Librarian</p> <p>University of Michigan Department of Chemical &amp; Metallurgical Engineering Ann Arbor, Michigan Attn: Librarian</p> <p>Vought Astronautics P. O. Box 5907 Dallas 22, Texas Attn: Librarian</p>	<p>Wah Chang Corporation Albany, Oregon Attn: Librarian</p> <p>Western Electric Company 222 Broadway New York 38, New York Attn: Librarian</p> <p>Western Electric Manufacturing Association 3600 Wilshire Boulevard Los Angeles 5, California Attn: Librarian</p> <p>Westinghouse Electric Corporation Astronuclear Laboratory P. O. Box 10864 Pittsburgh 36, Pennsylvania Attn: R. T. Begley</p>	<p>Westinghouse Electric Corporation Aerospace Electrical Division Lima, Ohio Attn: Librarian</p> <p>Westinghouse Electric Corporation Central Research Laboratory Pittsburgh 35, Pennsylvania</p> <p>Westinghouse Electric Corporation Research &amp; Development Laboratory Pittsburgh 35, Pennsylvania</p> <p>Wolverine Tube Division Calumet &amp; Hecla, Incorporated 17200 Southfield Road Allen Park, Michigan Attn: Mr. Eugene F. Hill</p>
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<p>Union Carbide Nuclear Company P. O. Box X Oak Ridge, Tennessee Attn: X-10 Laboratory Records Dept.</p> <p>United Nuclear Corporation Five New Street White Plains, New York Attn: Librarian</p> <p>University of Michigan Department of Chemical &amp; Metallurgical Engineering Ann Arbor, Michigan Attn: Librarian</p> <p>Vought Astronautics P. O. Box 5907 Dallas 22, Texas Attn: Librarian</p>	<p>Wah Chang Corporation Albany, Oregon Attn: Librarian</p> <p>Western Electric Company 222 Broadway New York 38, New York Attn: Librarian</p> <p>Western Electric Manufacturing Association 3600 Wilshire Boulevard Los Angeles 5, California Attn: Librarian</p> <p>Westinghouse Electric Corporation Astronuclear Laboratory P. O. Box 10864 Pittsburgh 36, Pennsylvania Attn: Librarian R. T. Begley</p>	<p>Westinghouse Electric Corporation Aerospace Electrical Division Lima, Ohio Attn: Librarian</p> <p>Westinghouse Electric Corporation Central Research Laboratory Pittsburgh 35, Pennsylvania</p> <p>Westinghouse Electric Corporation Research &amp; Development Laboratory Pittsburgh 35, Pennsylvania</p> <p>Wolverine Tube Division Calumet &amp; Hecla, Incorporated 17200 Southfield Road Allen Park, Michigan Attn: Mr. Eugene F. Hill</p>
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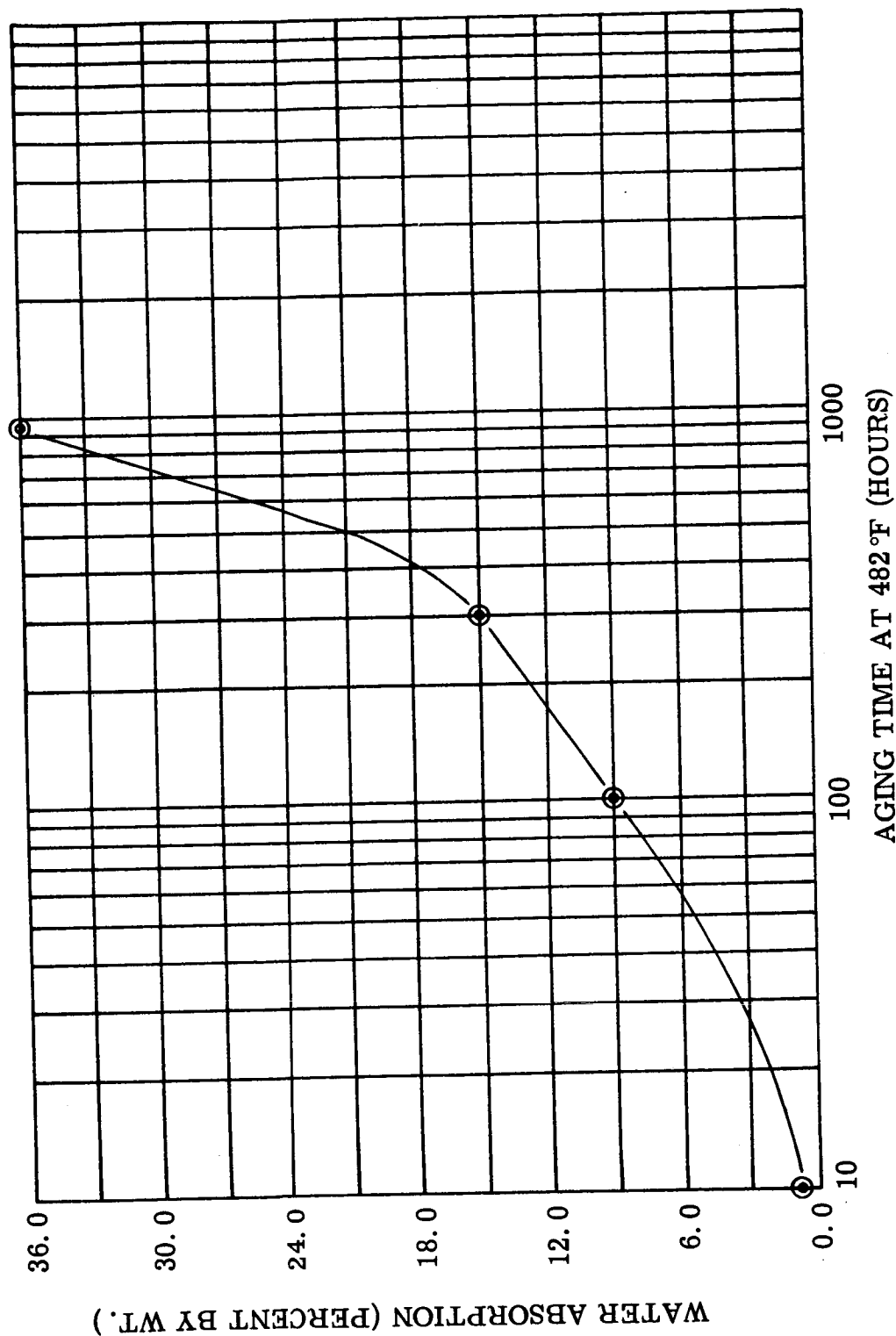


FIGURE IV-1. Water Absorption, Room Temperature Tests After Air Aging, Micarta  
H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)

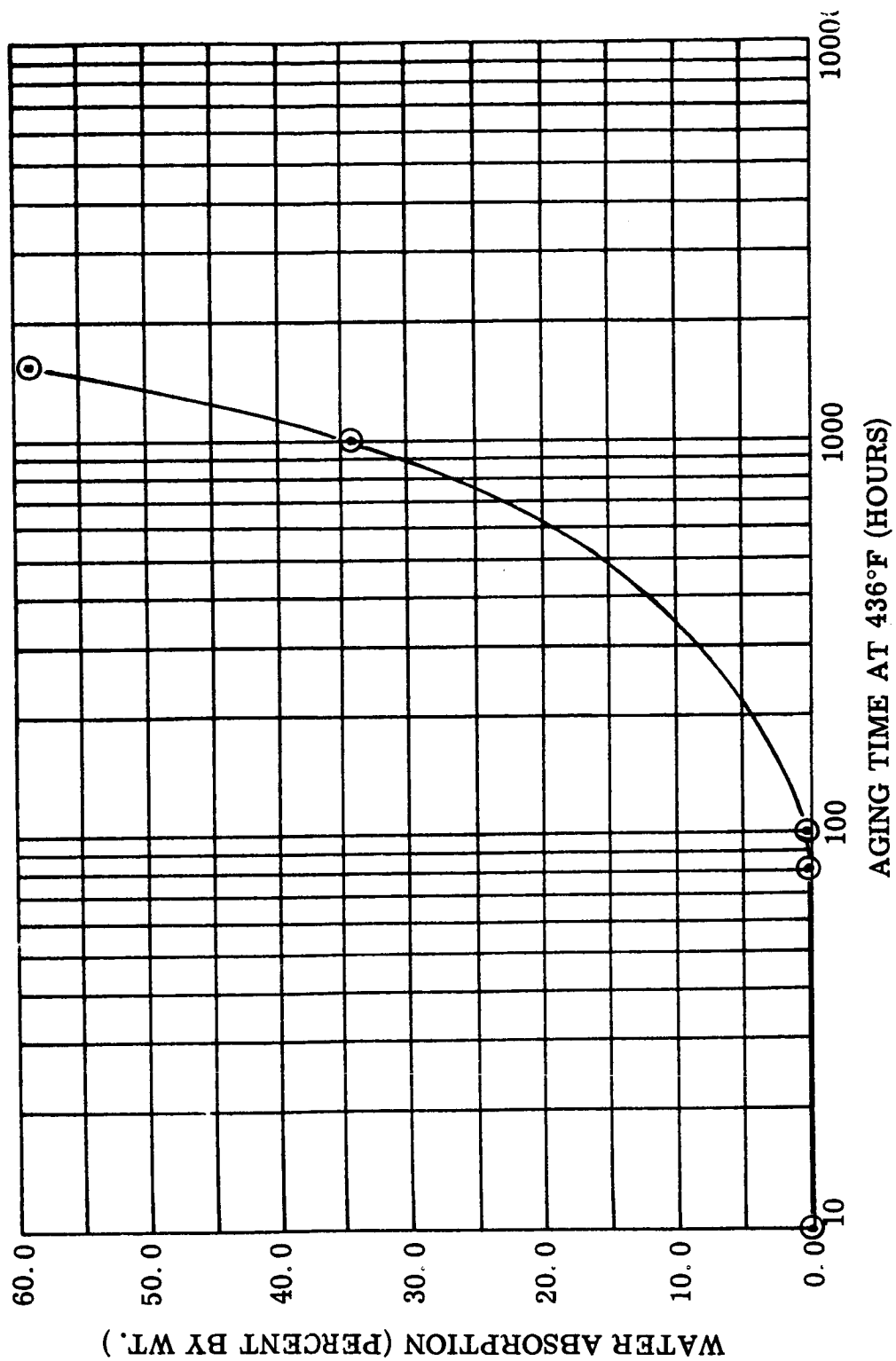
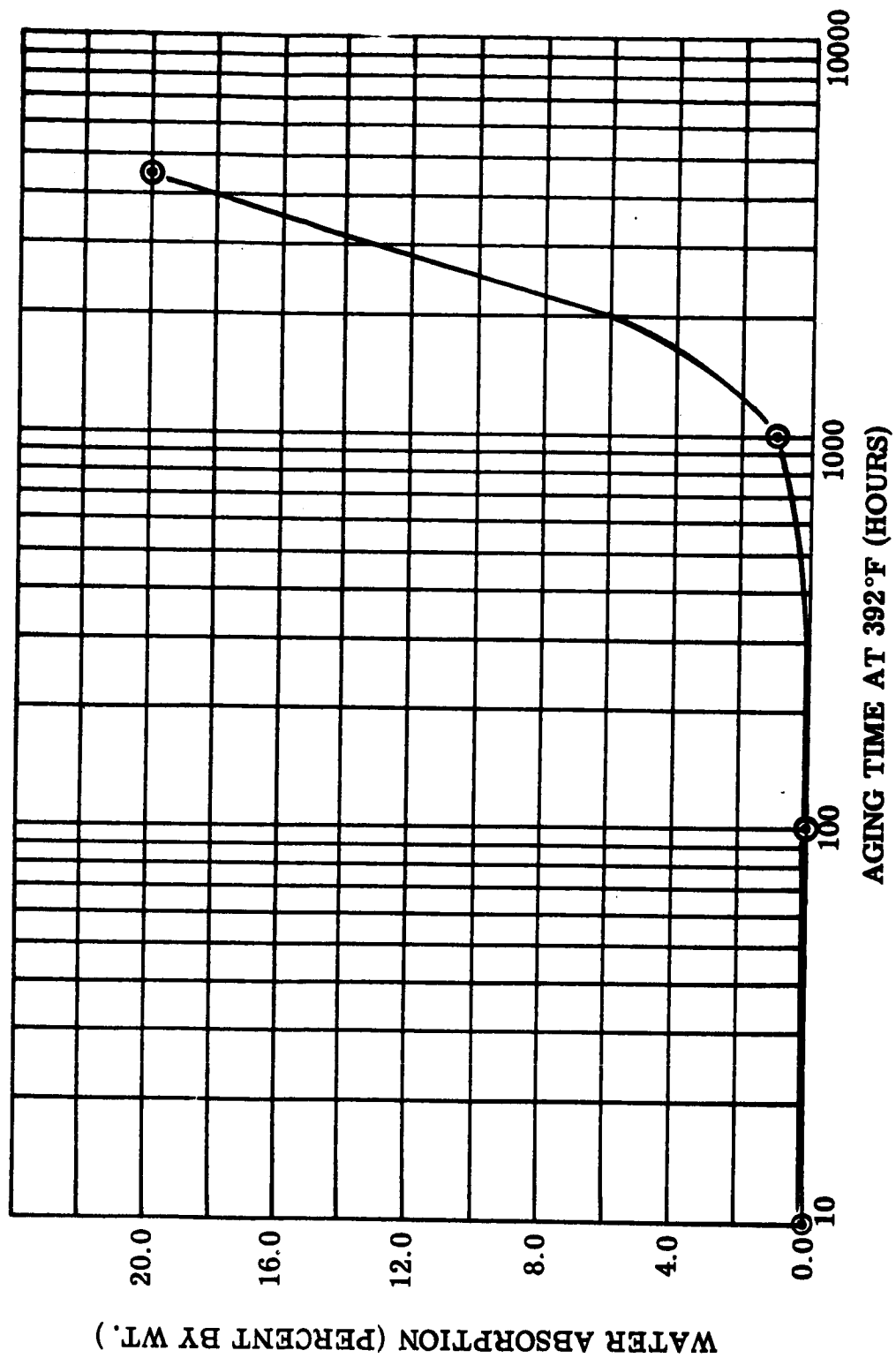
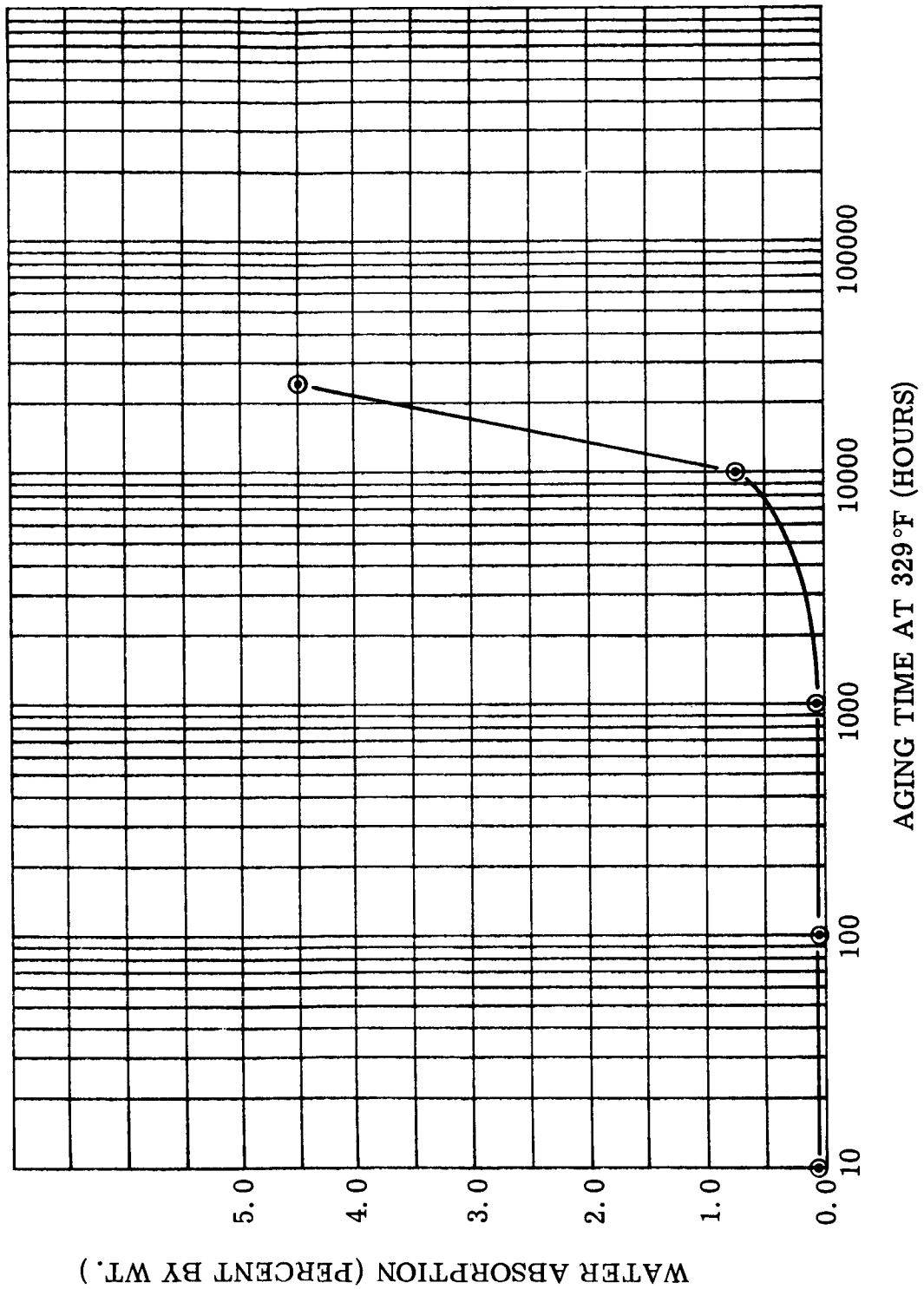


FIGURE I V-2. Water Absorption, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Water Absorption - Epoxy Glass Laminate

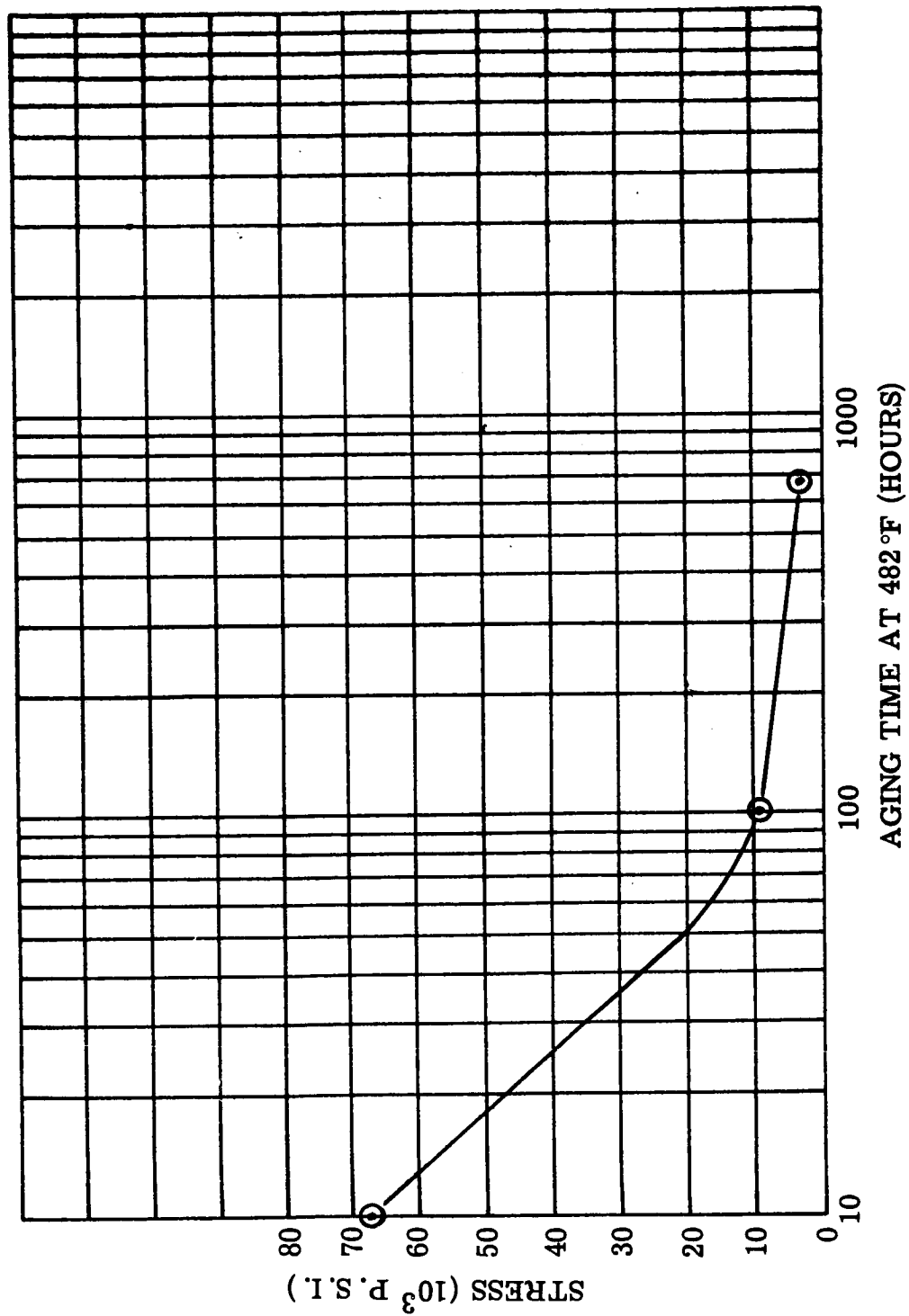
FIGURE IV-3. Water Absorption, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Water Absorption - Epoxy Glass Laminate

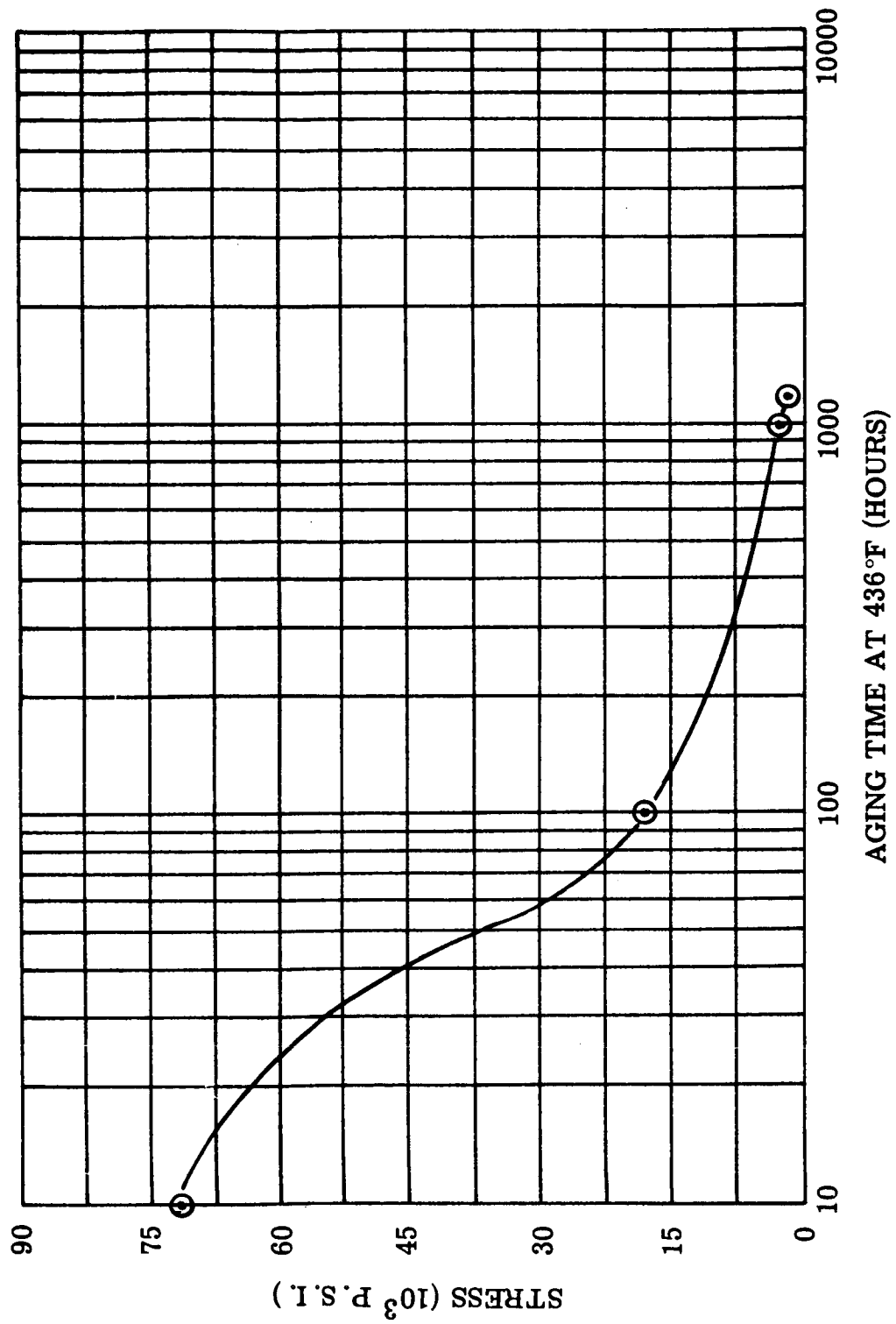
WAED64. 14E-86

FIGURE IV-4. Water Absorption, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Aged Flexural Strength - Epoxy Glass Laminate

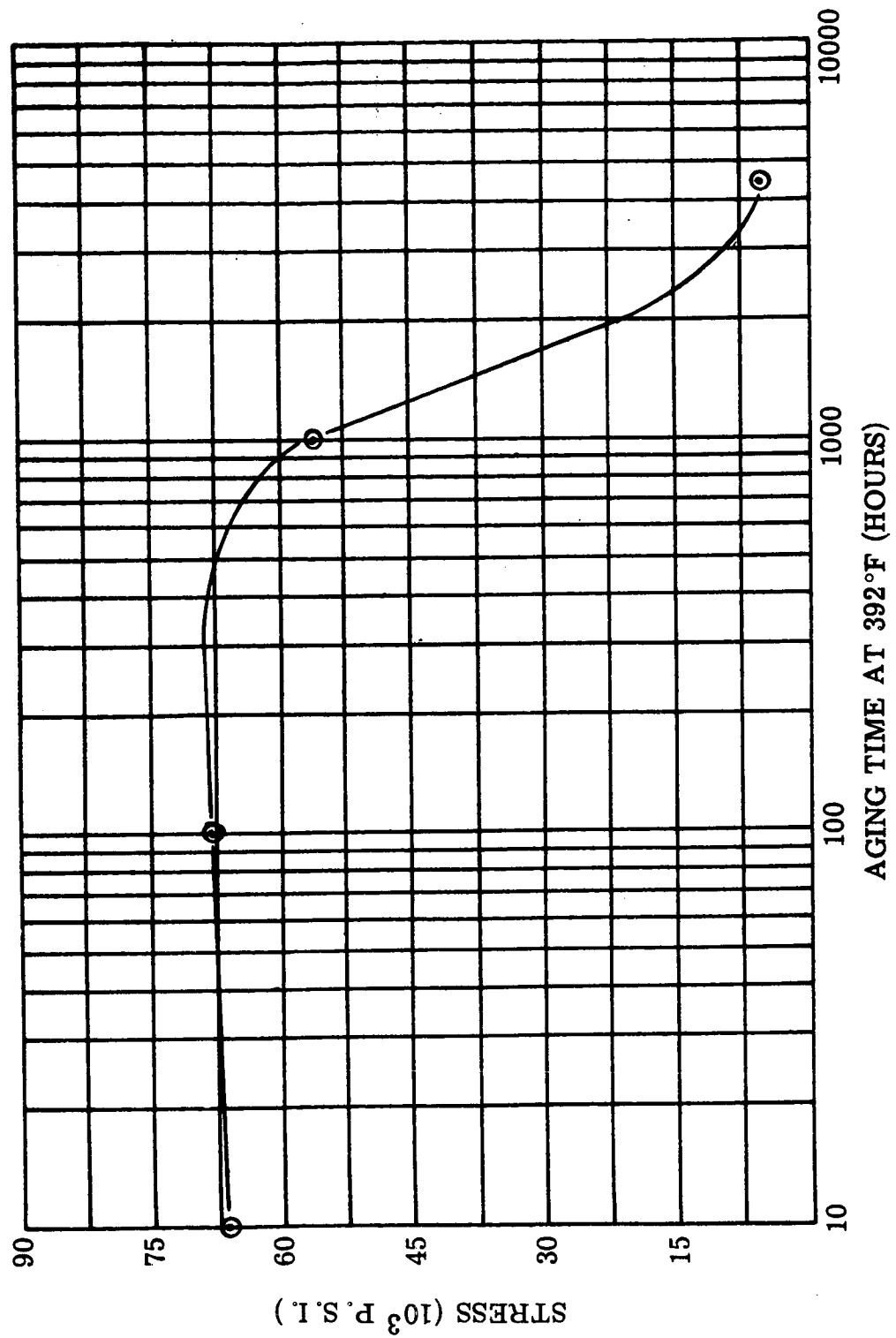
FIGURE IV-5. Flexural Strength, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Aged Flexural Strength - Epoxy Glass Laminate

FIGURE IV-6. Flexural Strength, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)





Aged Flexural Strength - Epoxy Glass Laminate

FIGURE IV-7. Flexural Strength, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)

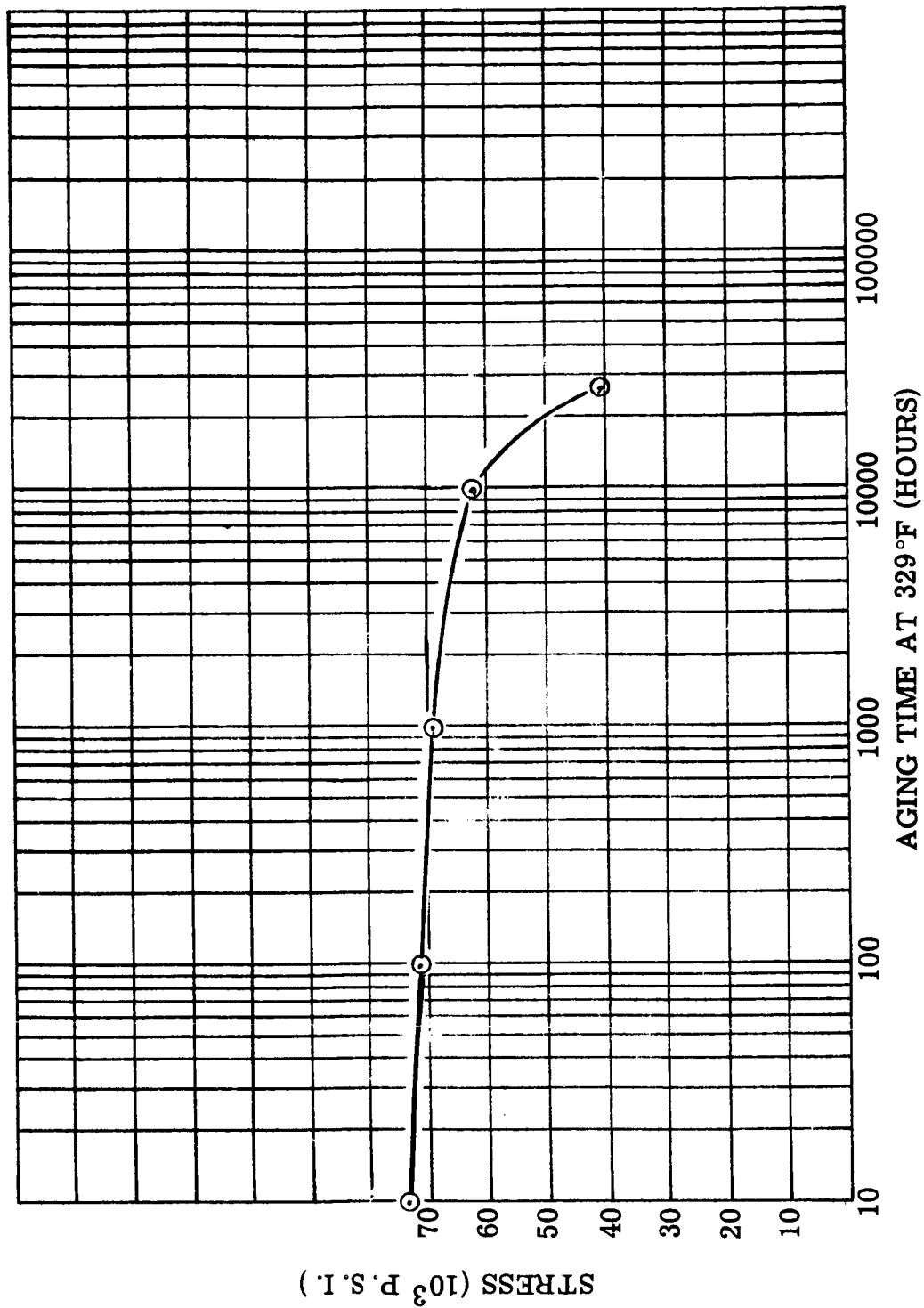
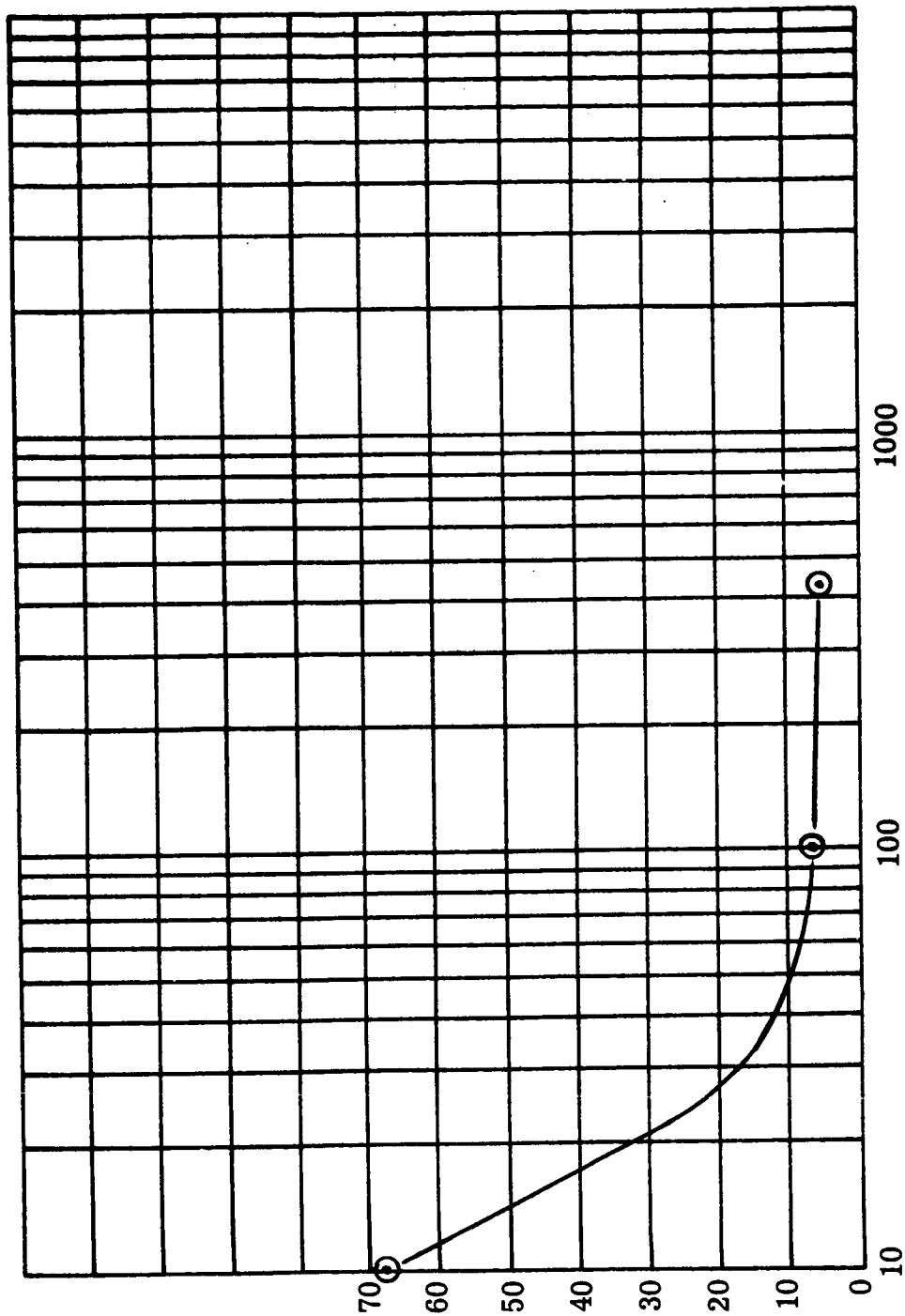


FIGURE I V-8. Flexural Strength, Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)

Aged Flexural Strength - Epoxy Glass Laminate

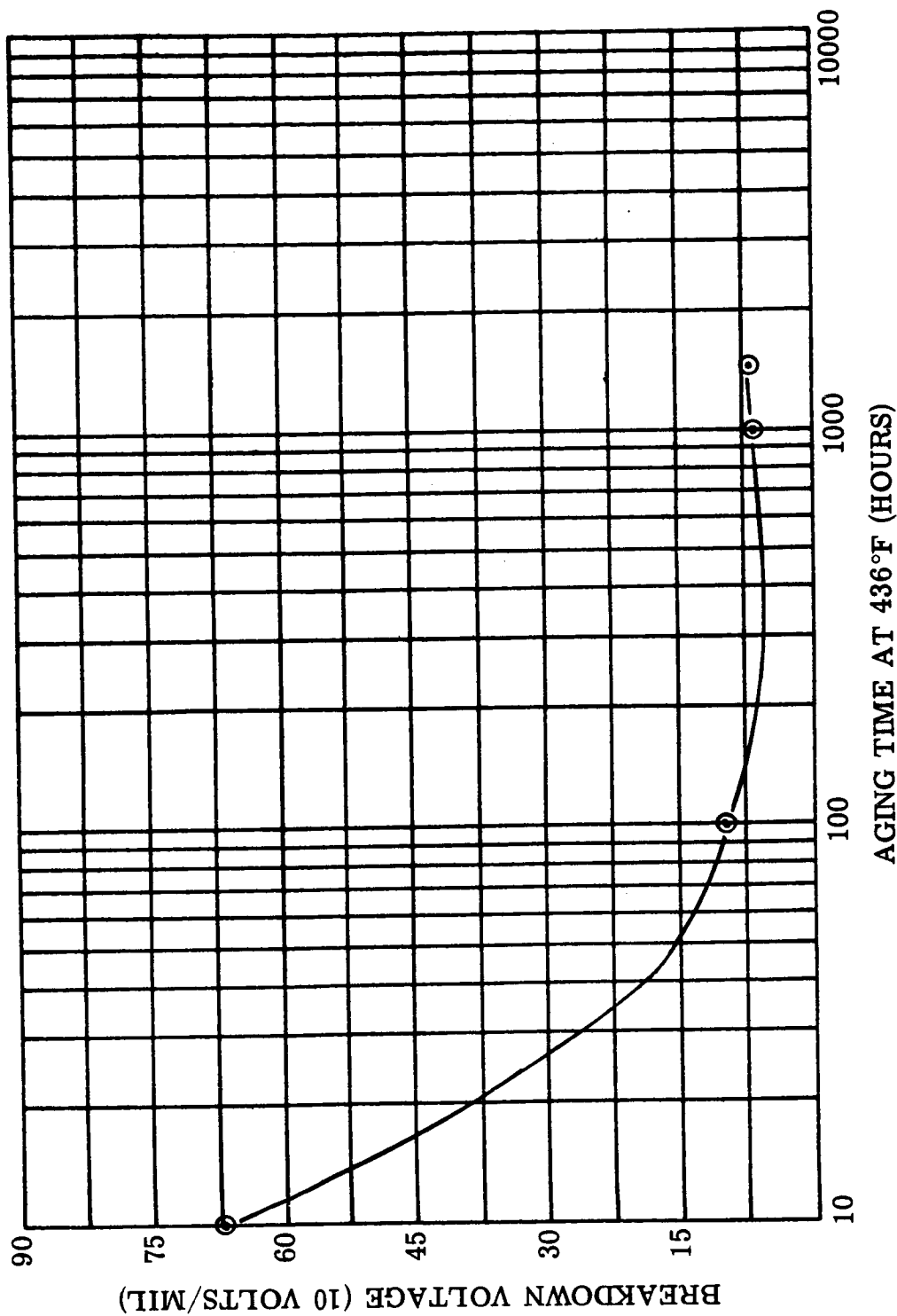
BREAKDOWN VOLTAGE (10 VOLTS/MIL)

Insulation Life - Epoxy Glass Laminate



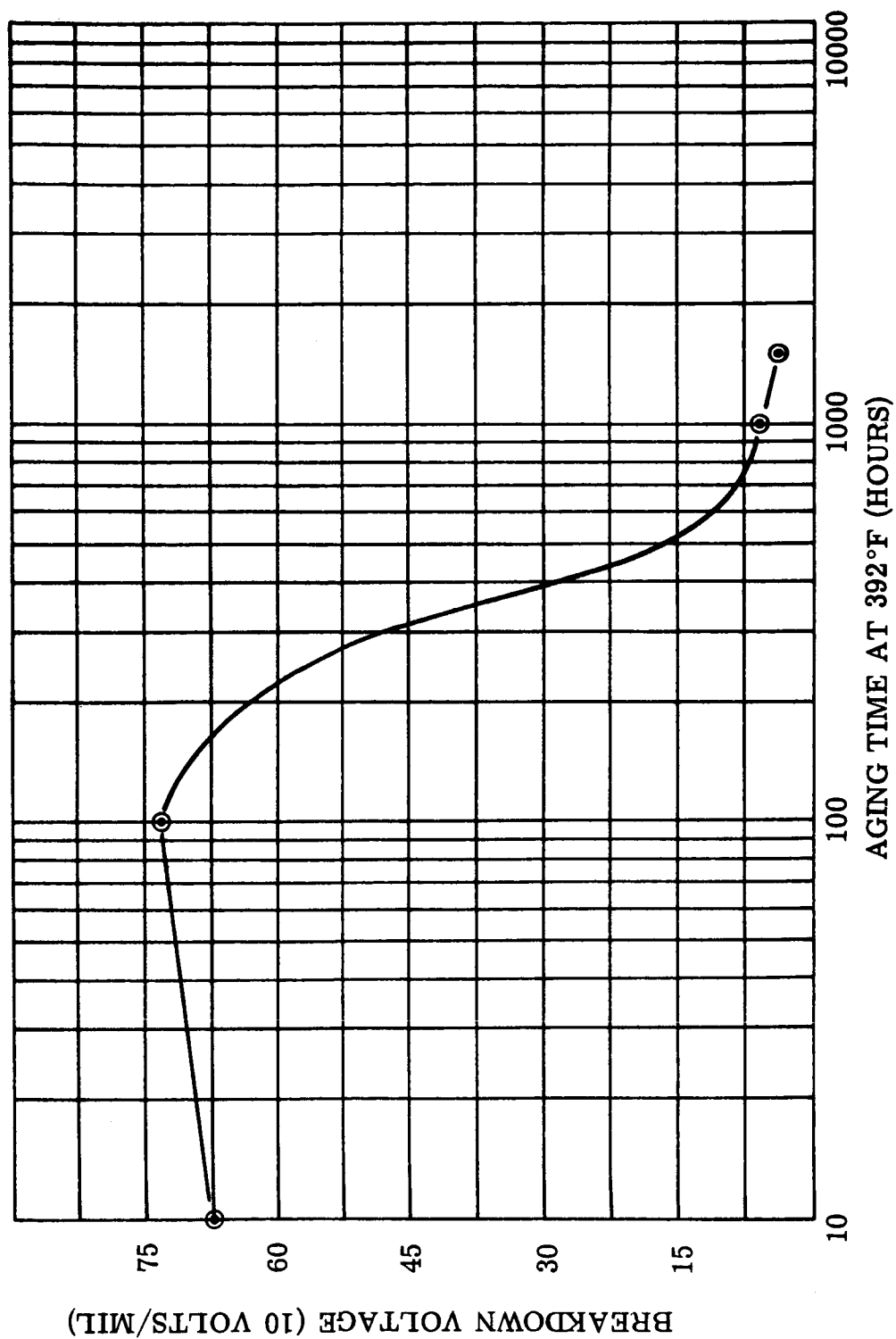
AGING TIME 482°F (HOURS)

FIGURE I V-9. Insulation Life (Electric Strength), Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



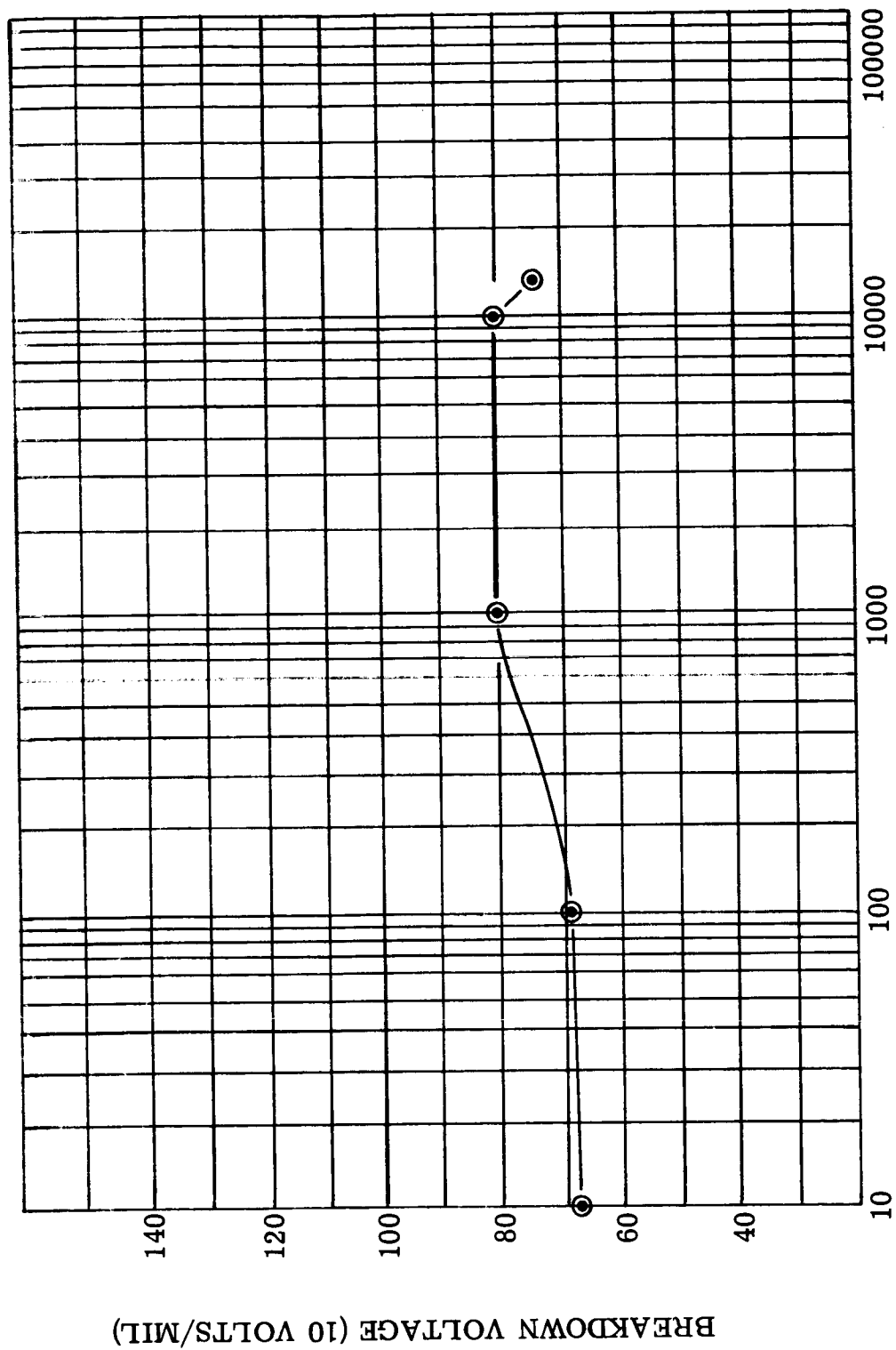
Insulation Life - Epoxy Glass Laminate

FIGURE IV-10. Insulation Life (Electric Strength), Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Insulation Life - Epoxy Glass Laminate

FIGURE I V-11. Insulation Life (Electric Strength), Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)



Insulation Life - Epoxy Glass Laminate

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FIGURE I V-12. Insulation Life (Electric Strength), Room Temperature Tests After Air Aging, Micarta H-2497 Epoxy Glass Reinforced Laminate (Reference: RI252)

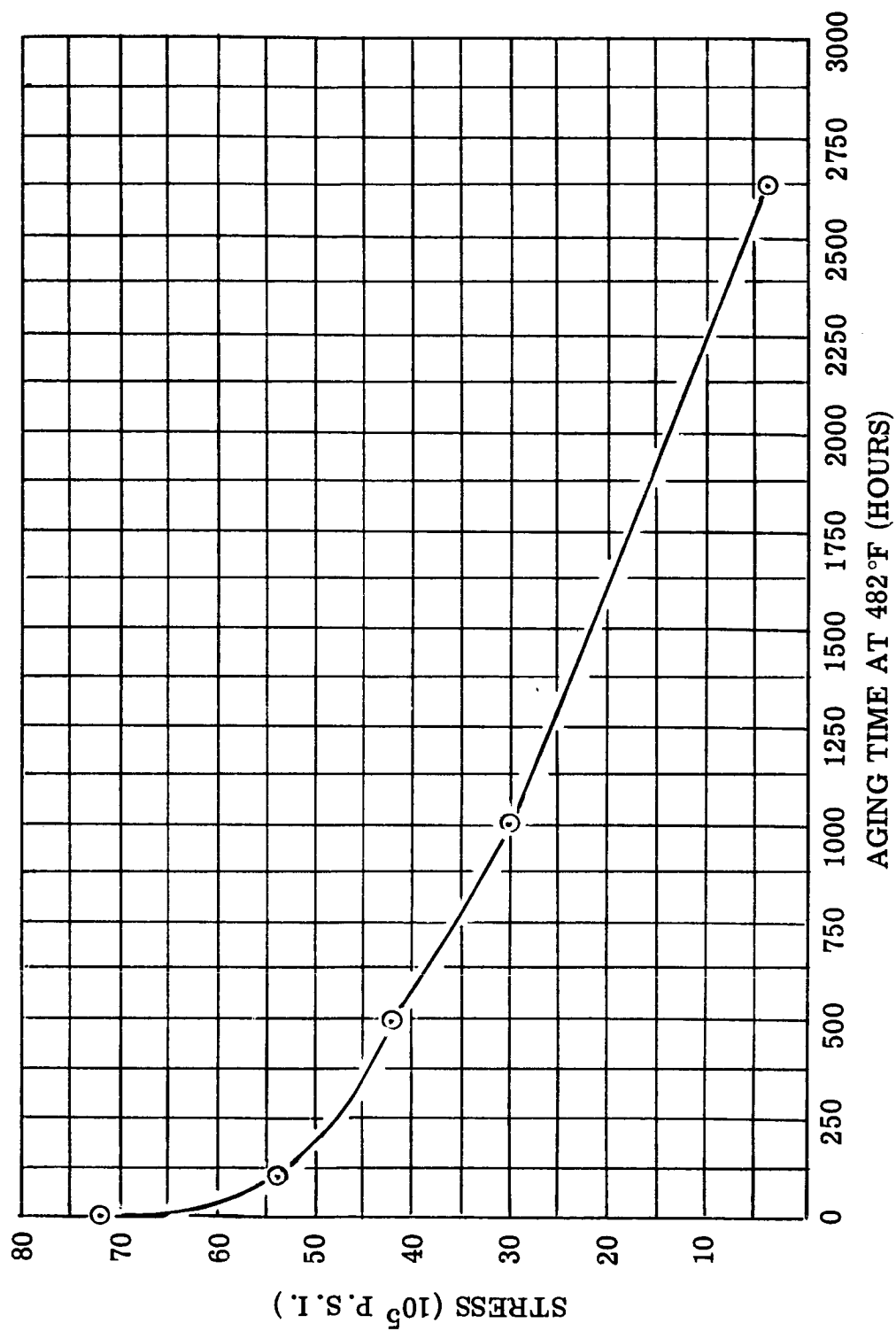


FIGURE I V-13. Endurance Strength (Flexural Strength), Room Temperature Tests After Air Aging, Diphenyl Oxide Laminate Grade H 17511, Tested at 73°F (Reference: RI217)

Aged Flexural Strength at Temperature - Diphenyl Oxide Laminate

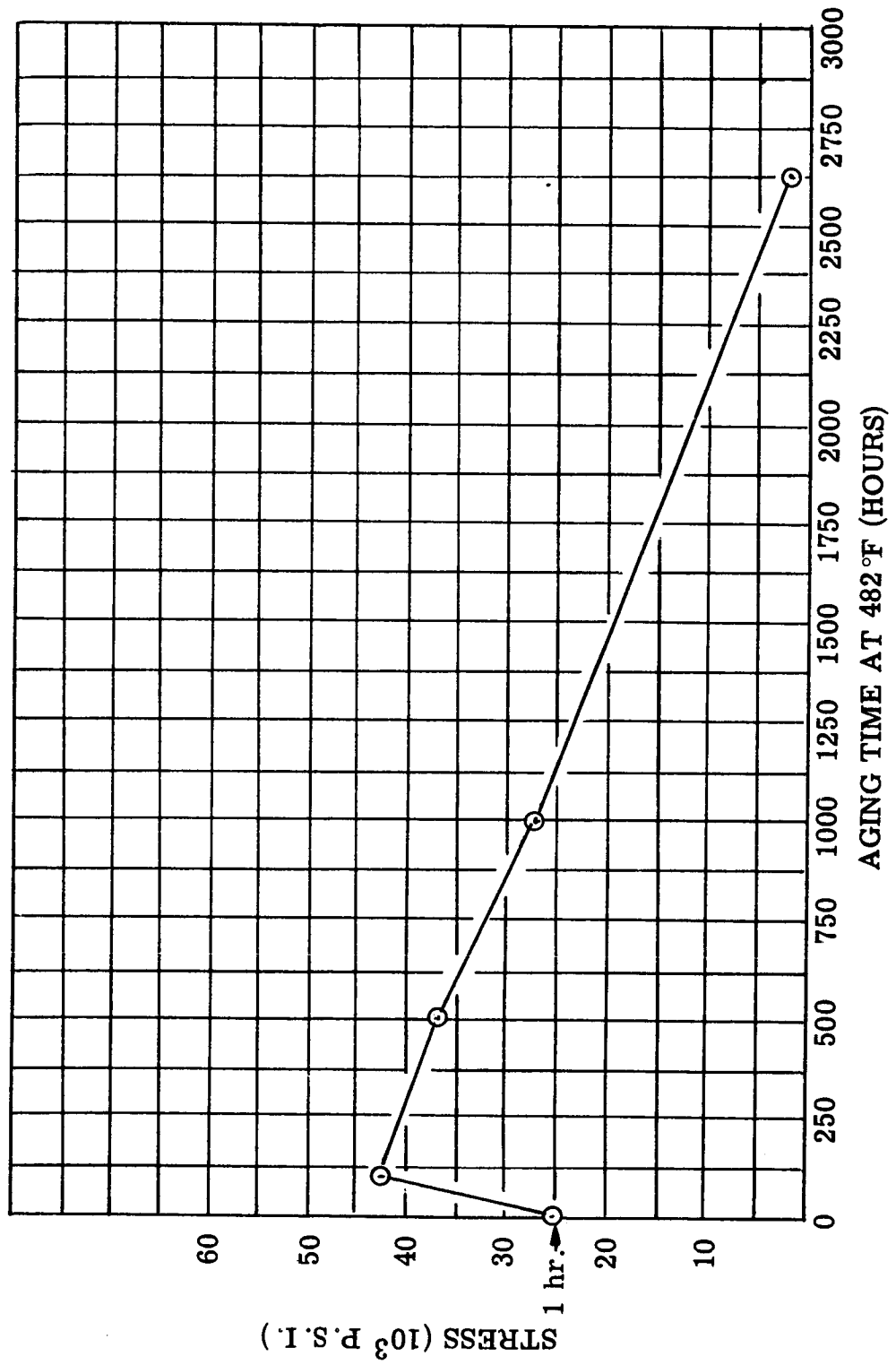


FIGURE IV-14. Flexural Strength, Elevated Temperature Tests After Air Aging, Diphenyl Oxide Laminate Grade H 17511, Tested at 482°F (Reference: RI217)



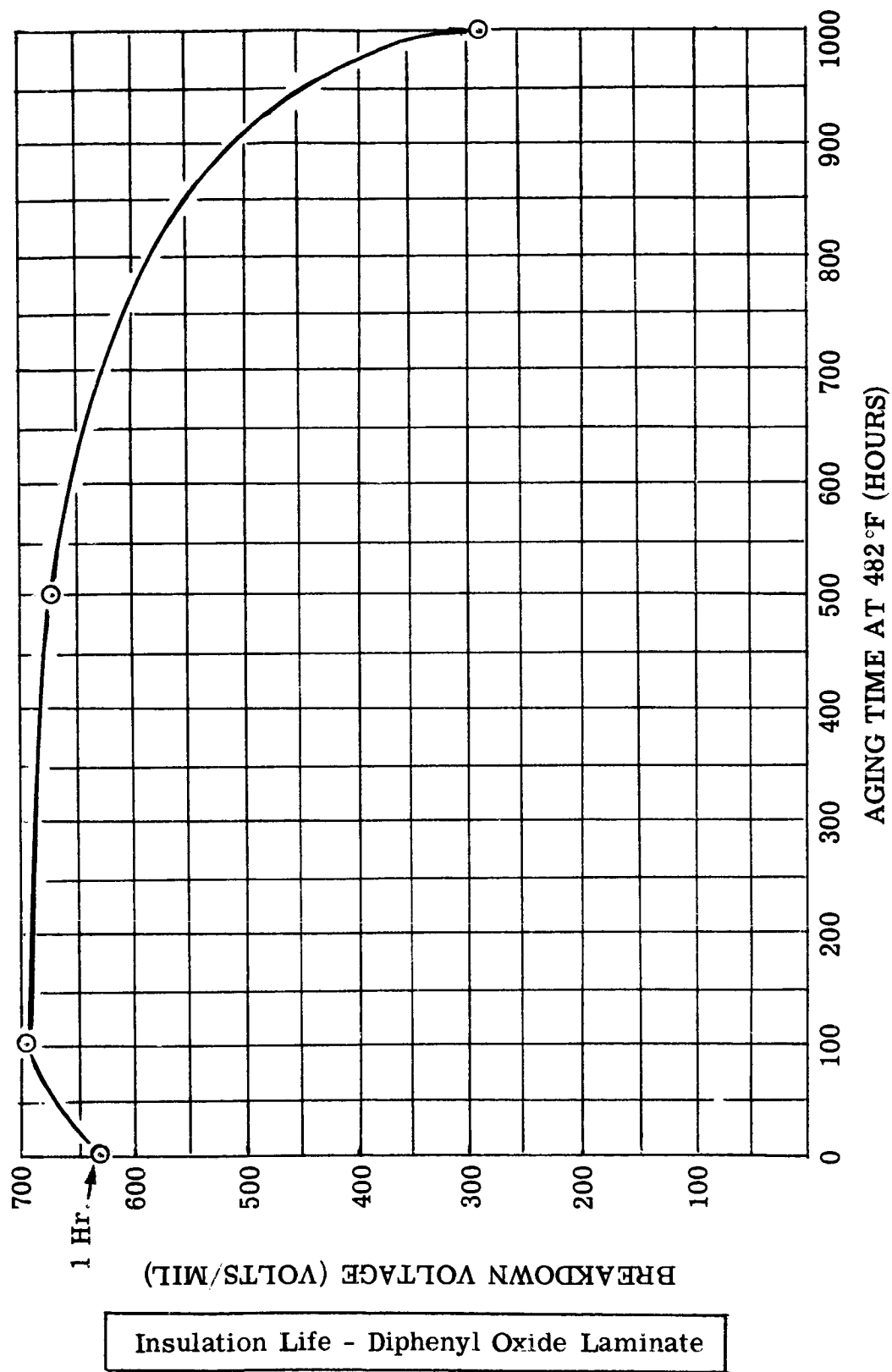
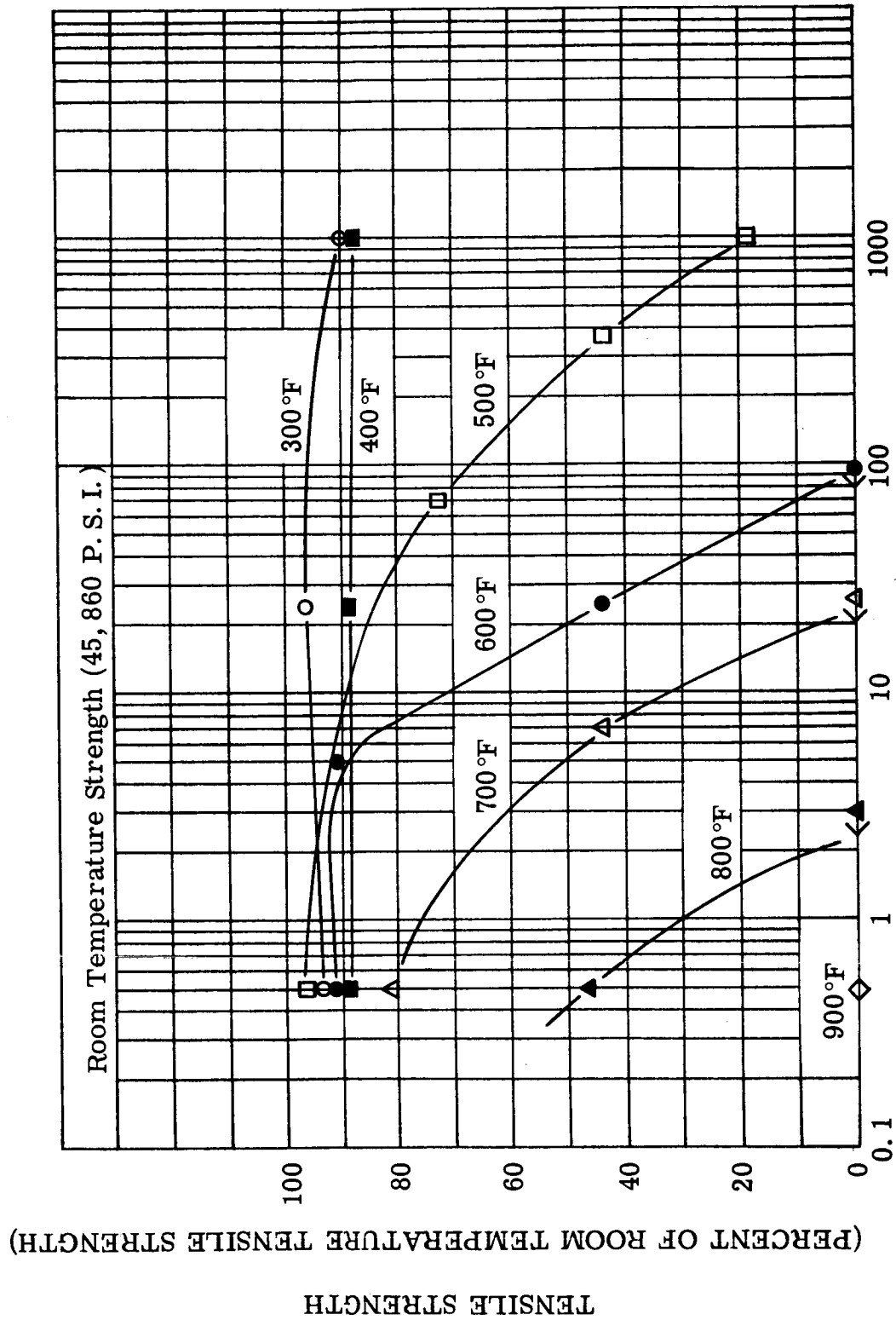


FIGURE I V-15. Insulation Life (Electric Strength), Room Temperature Tests After Air Aging, Diphenyl Oxide Laminate Grade H 17511, Tested at 73°F (Reference: RI217)

Tensile Strength vs Temperature - Phenolic Glass Laminate

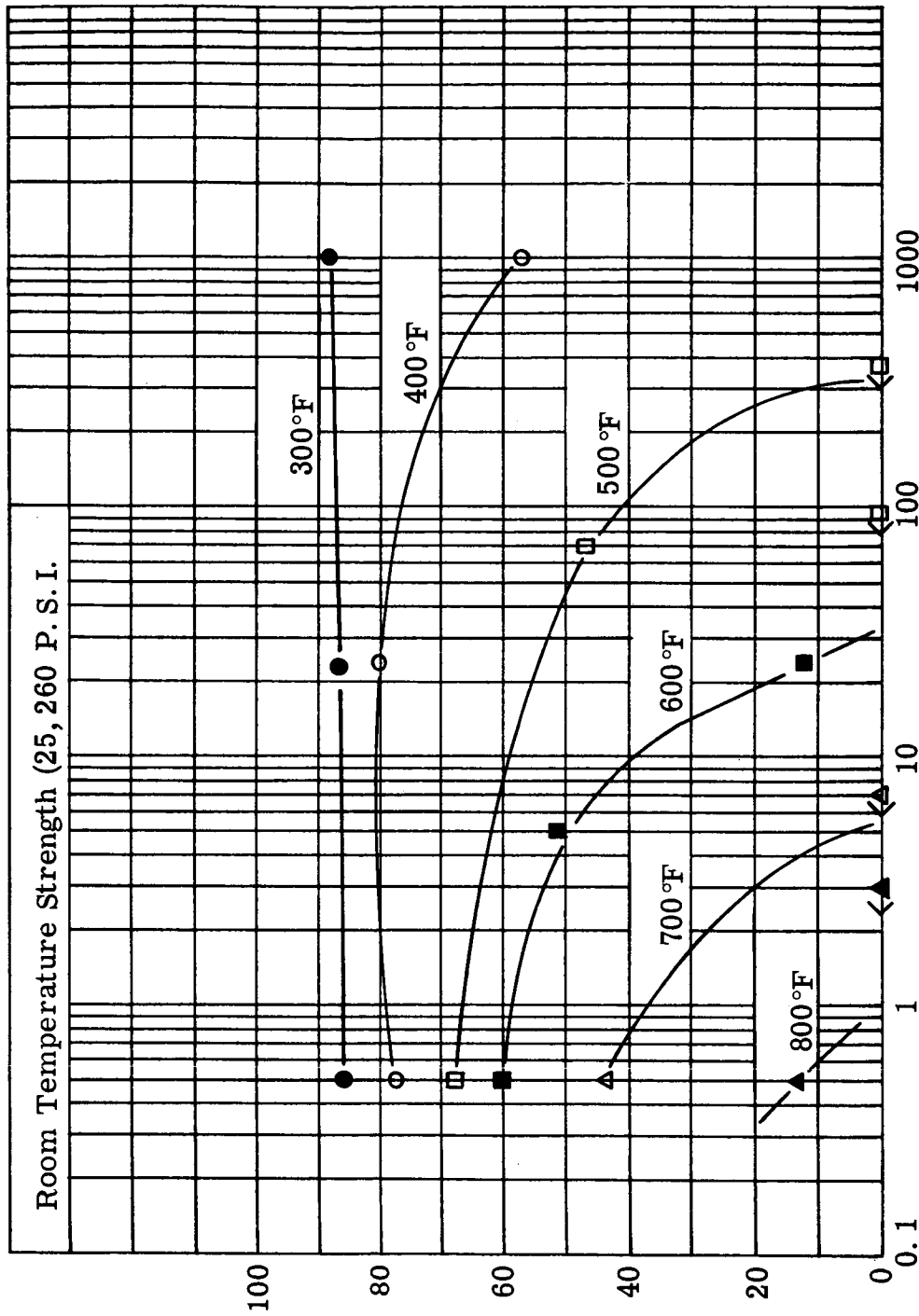


EXPOSURE TIME TO TEST TEMPERATURES (HOURS)

FIGURE I V-16. Tensile Strength in Air, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RI516)

Tensile Strength (45°  $\Delta$ ) vs Temperature - Phenolic Glass Laminate

TENSILE STRENGTH  
(PERCENT OF ROOM TEMPERATURE TENSILE STRENGTH)



EXPOSURE TIME TO TEST TEMPERATURES (HOURS)

FIGURE I V-17. Tensile Strength in Air at 45° angle to Warp, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RI516)

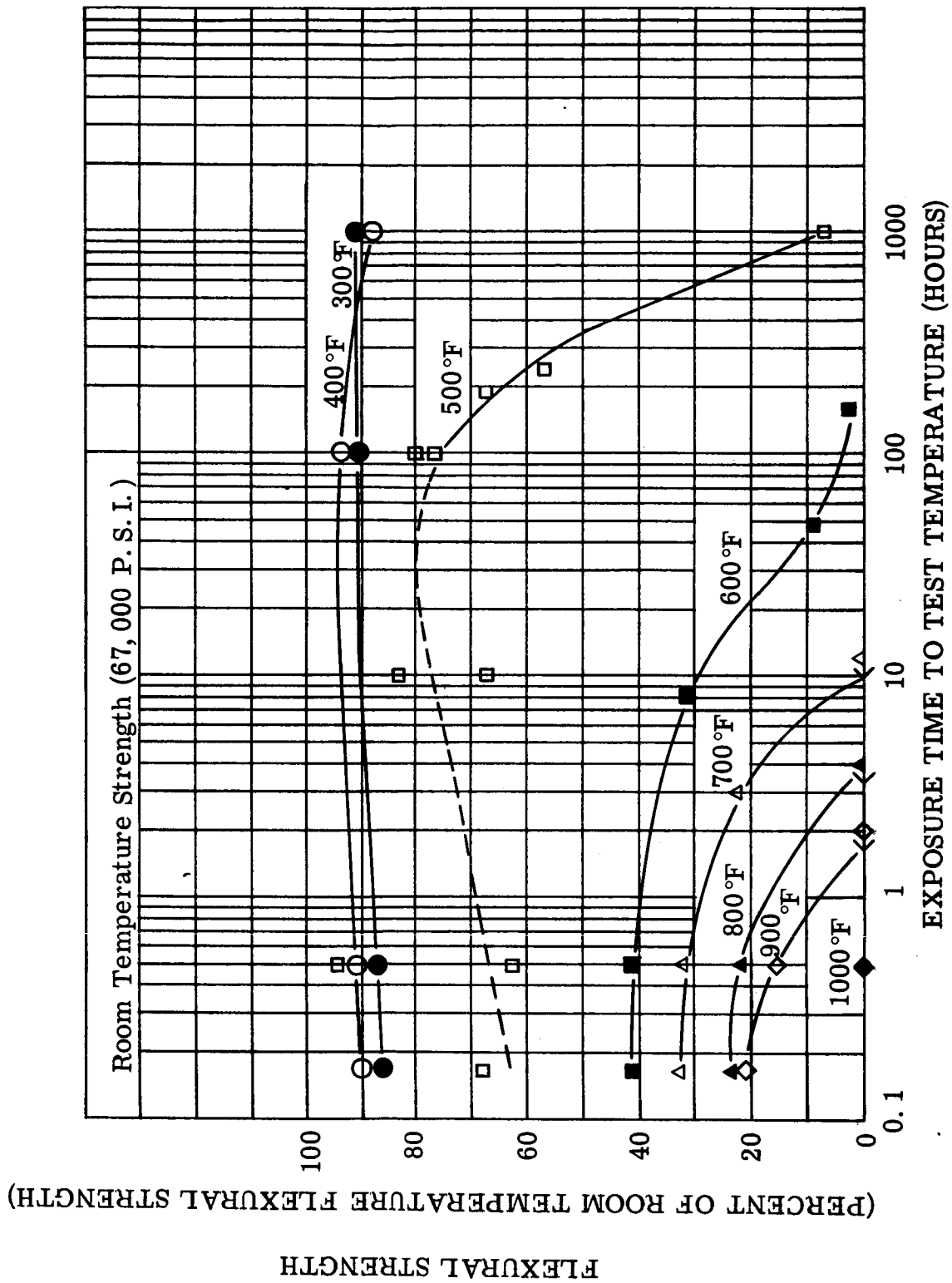


FIGURE IV-18. Flexural Strength in Air, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RI516)

# Compressive Strength - Phenolic Glass Laminate

(PERCENT OF ROOM TEMPERATURE COMPRESSIVE STRENGTH)

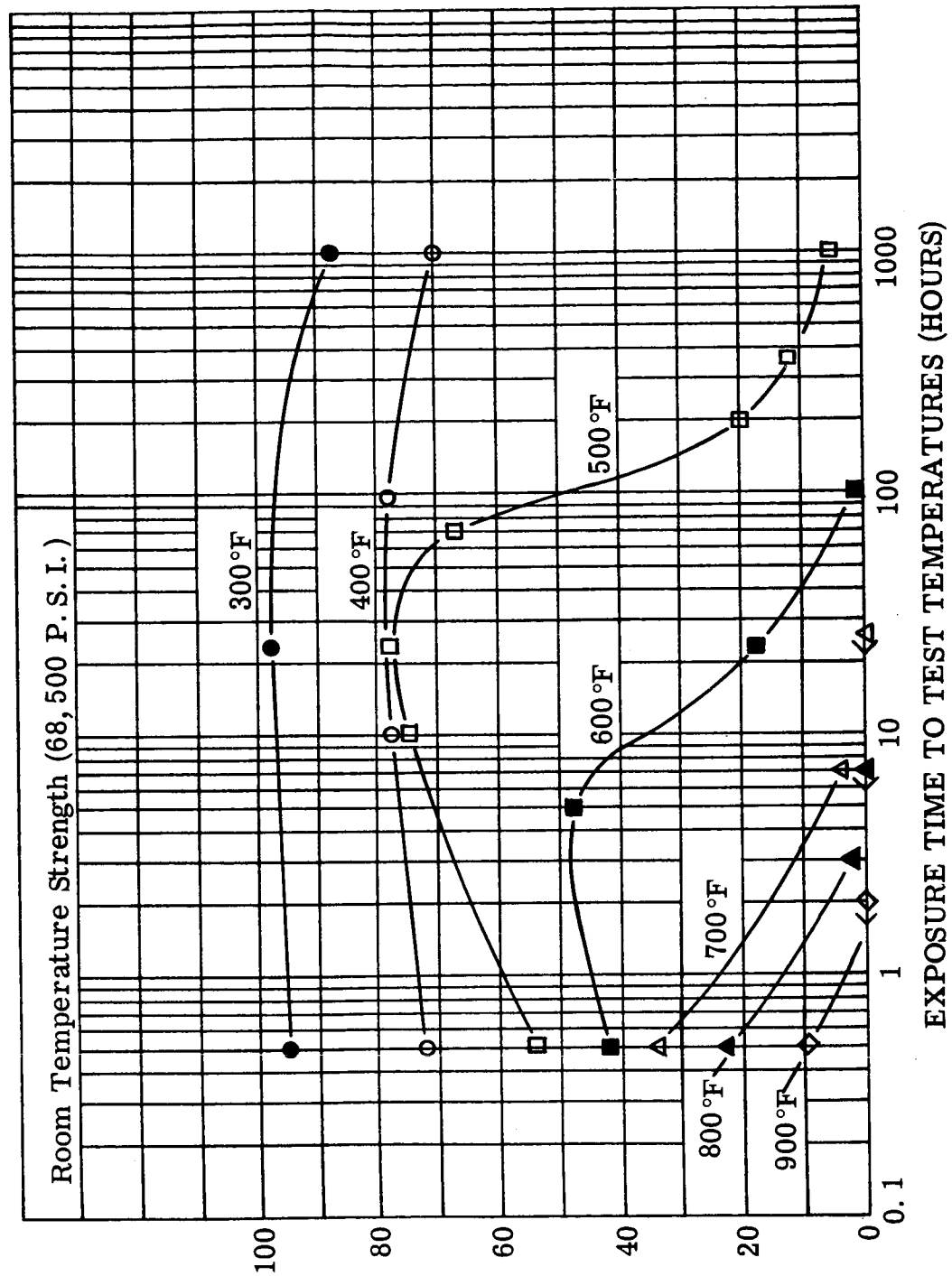


FIGURE IV-19. Compressive Strength in Air, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RI516)

# Flexural Modulus vs Temperature - Phenolic Glass Laminate

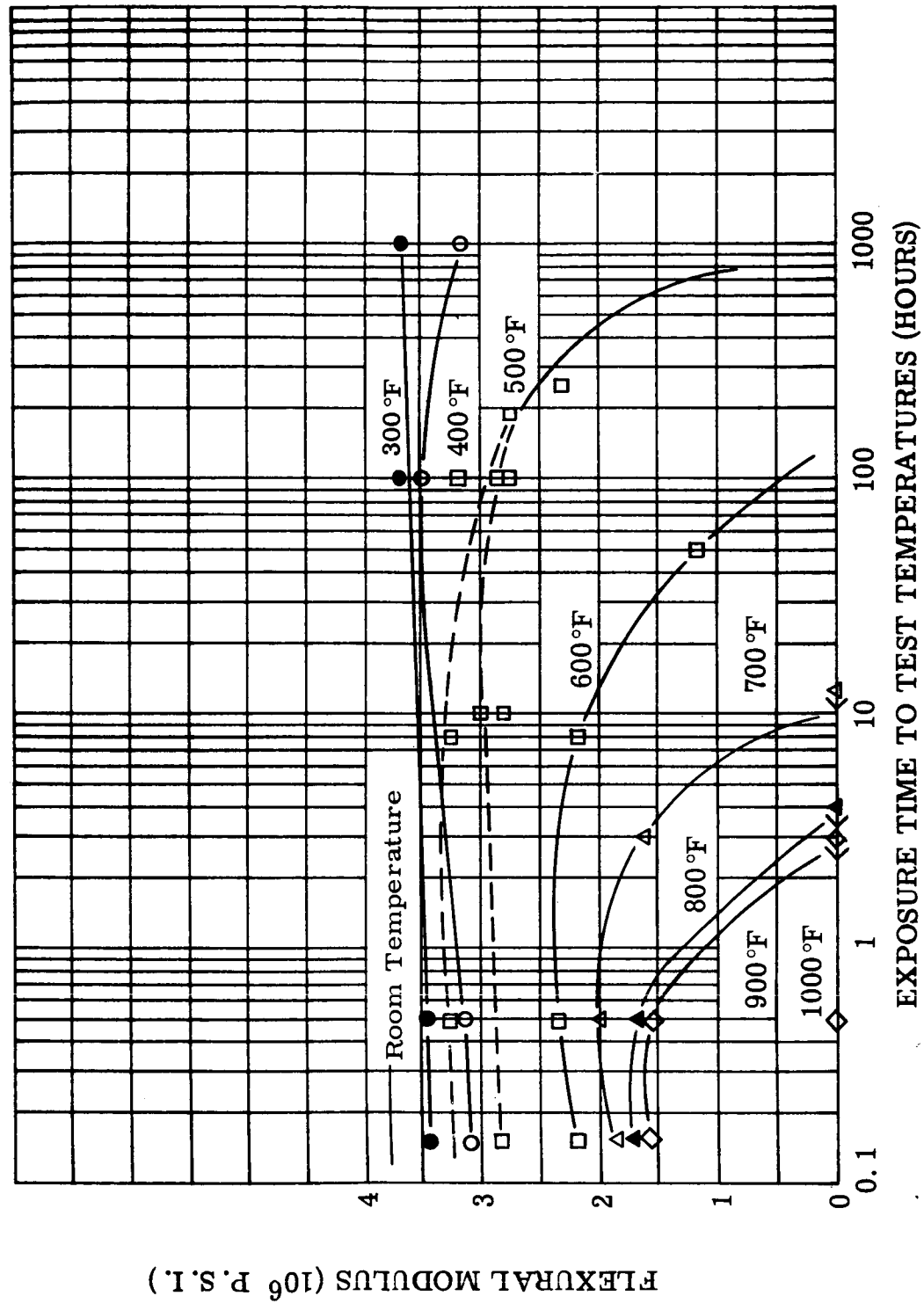
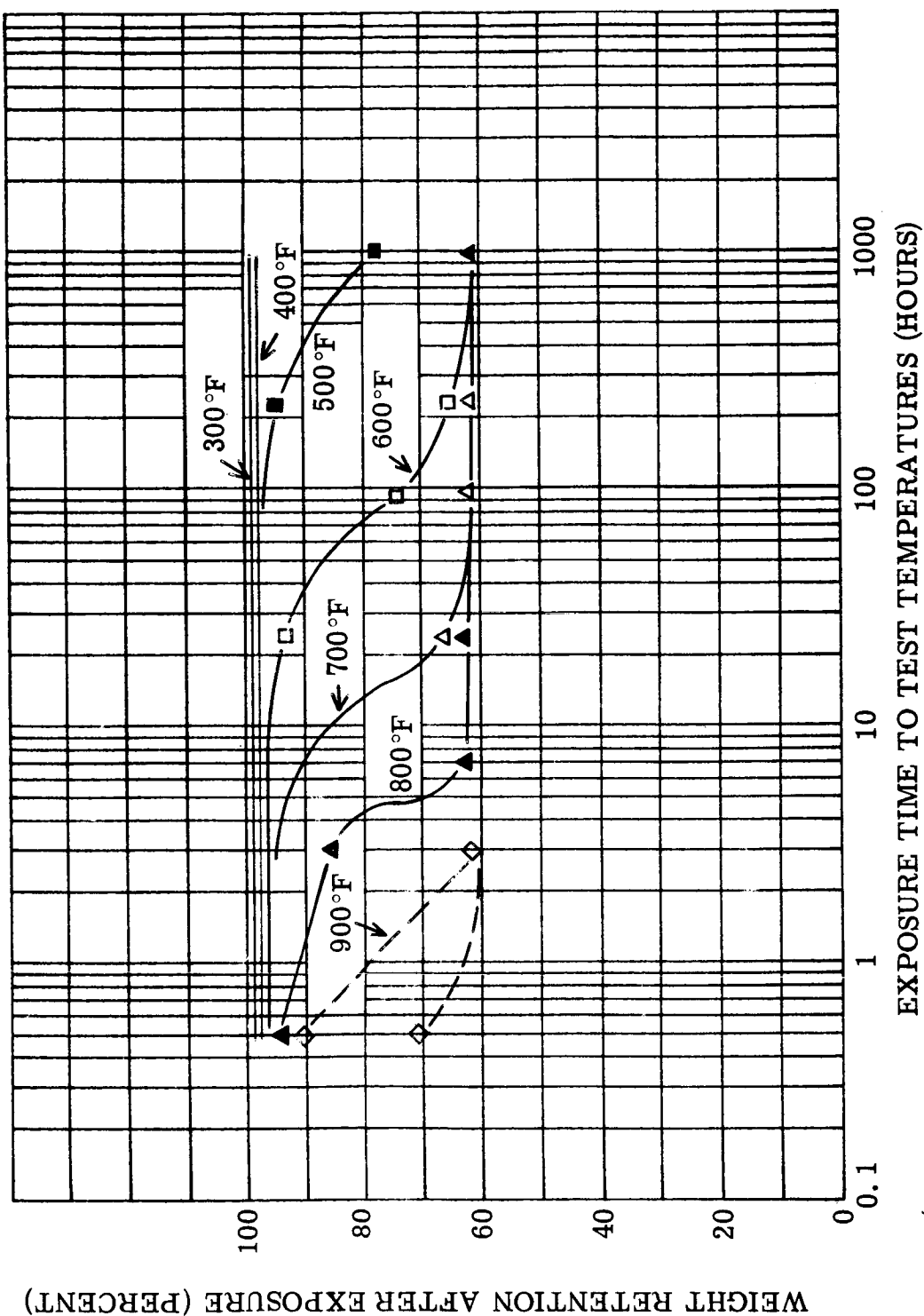


FIGURE I V-20. Flexural Modulus in Air, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RJ516)



Weight Loss - Phenolic Glass Laminate

FIGURE IV-21. Deterioration as Weight Loss in Air, CTL-91 LD Resin Phenolic Glass Fabric Laminate (181 A1100)(Reference: RI516)

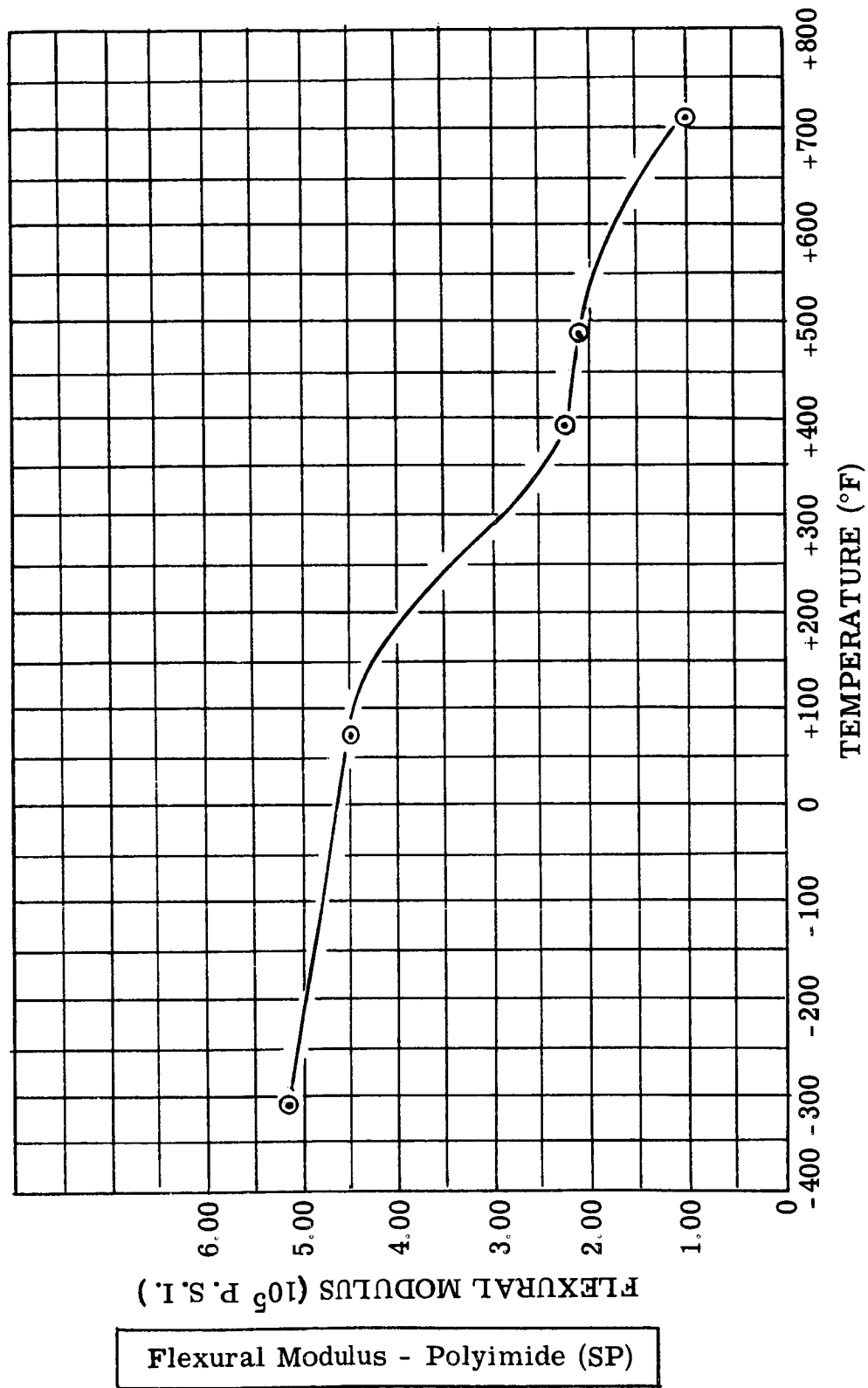


FIGURE IV-22. Flexural Modulus in Air, Polyimide Molding Compound (SP) (Reference: RI516)



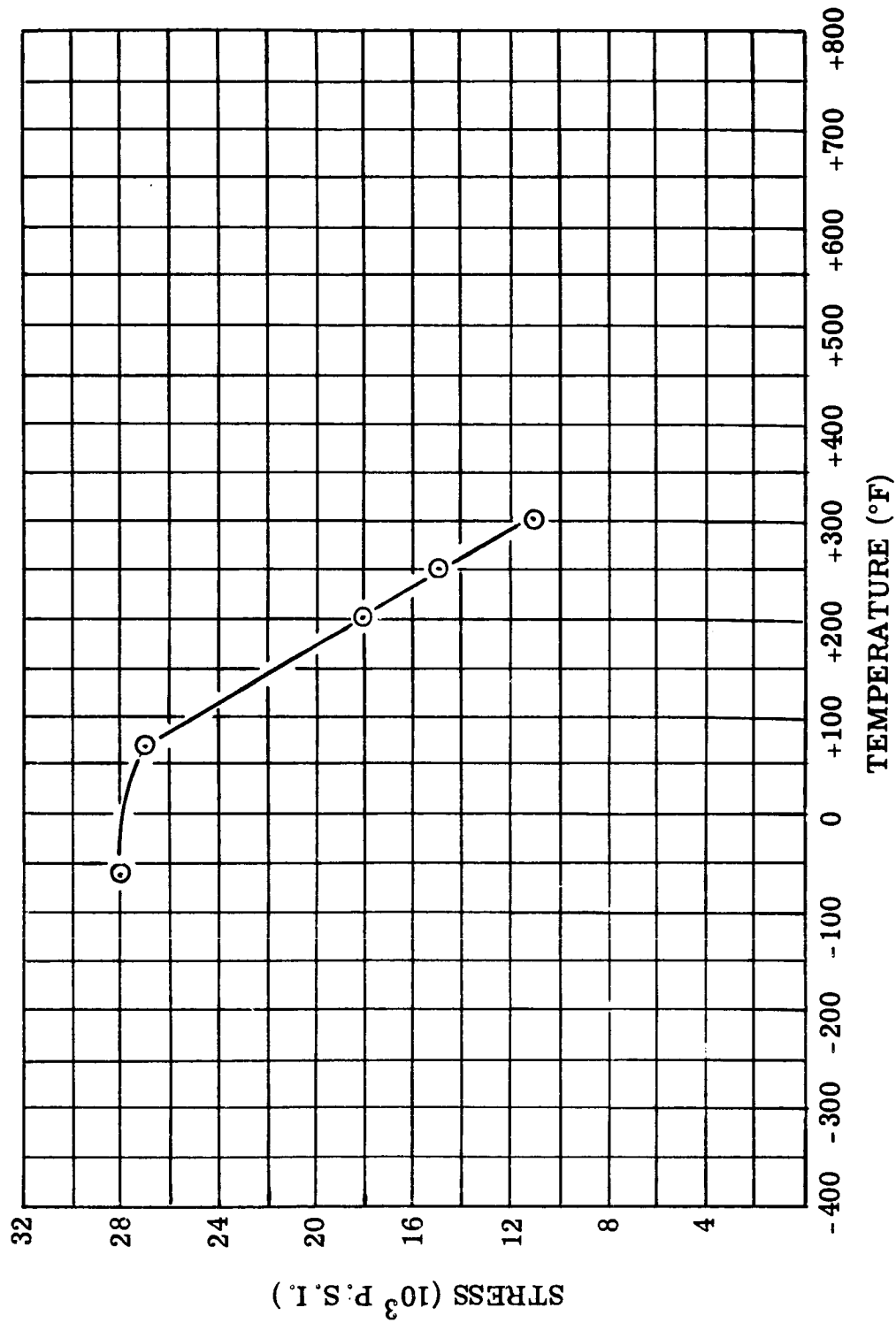


FIGURE IV-23. Tensile Strength in Air, Scotchply Type 1100 HSMC, Aged 0.5 Hours at Temperature (Reference: RI160)

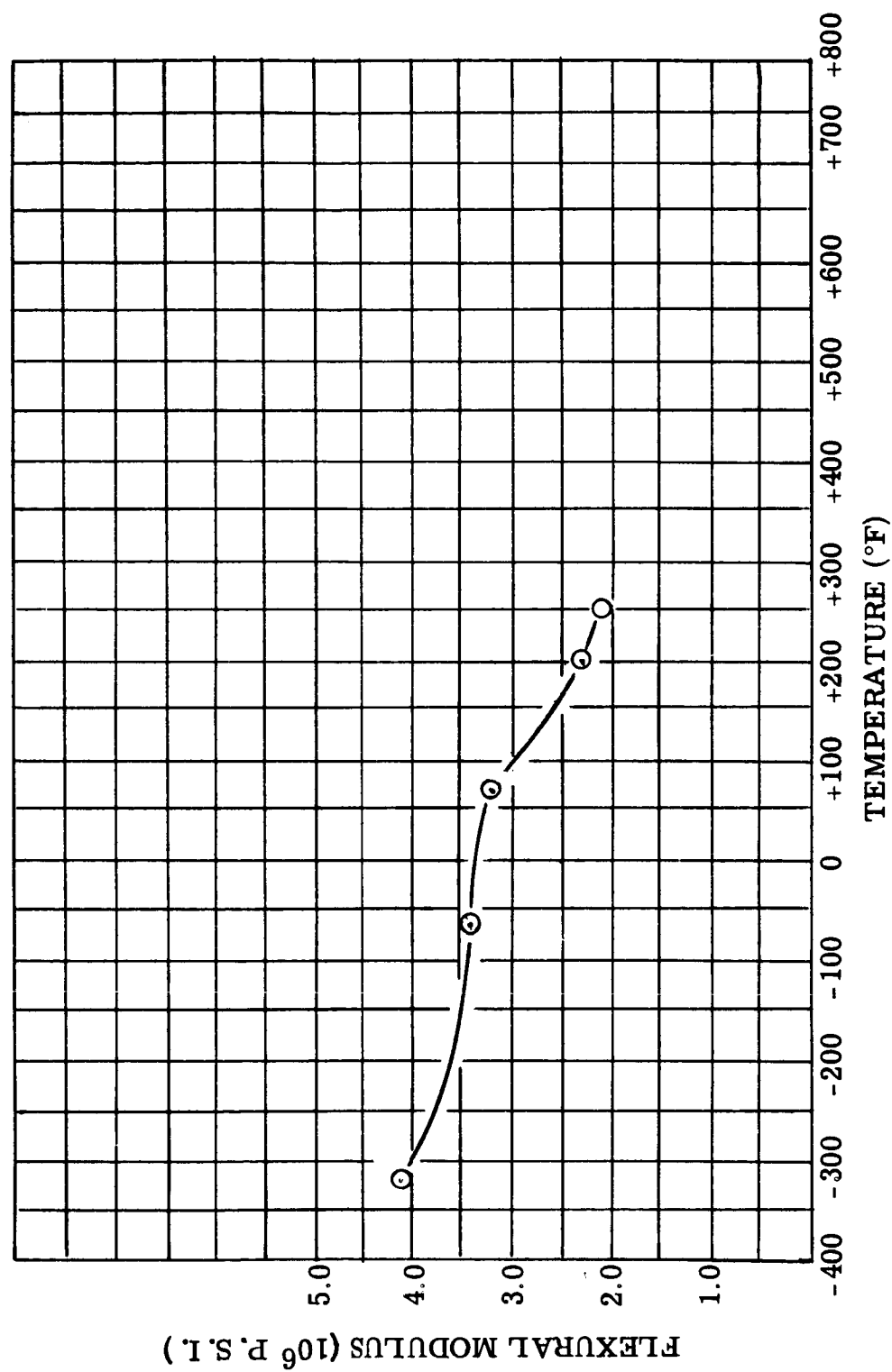


FIGURE IV-24. Modulus in Flexure in Air, Scotchply Type 1100 HSMC, Aged 0.5 Hours at Temperature (Reference: RI160)

Flexural Modulus - Epoxy Premix

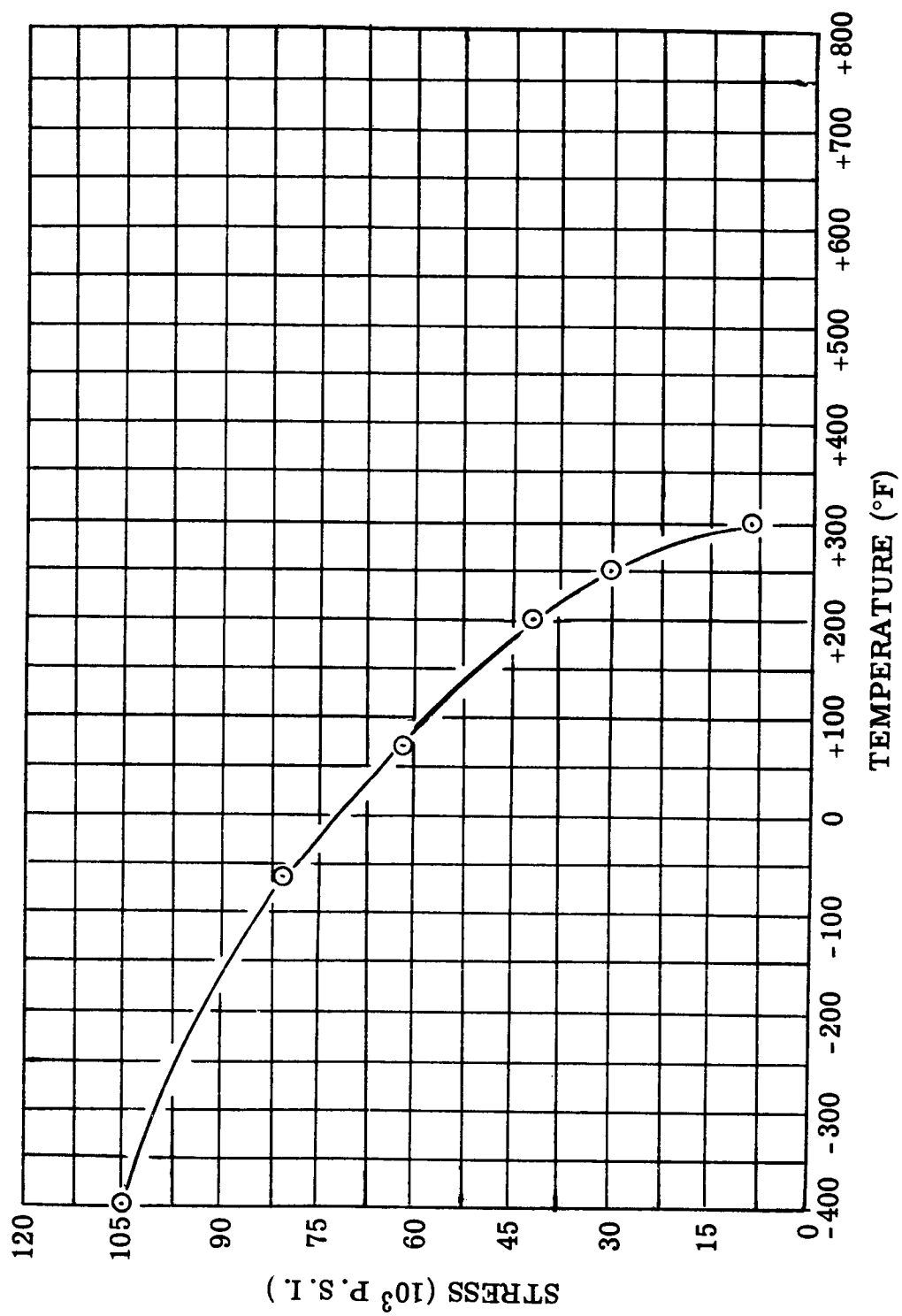


FIGURE I V-25. Flexural Strength in Air, Scotchply Type 1100 HSMC Aged 0.5 Hours at Temperature (Reference):

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- RI123 Freeman, J. H. , Private Correspondence J. Freeman to C. Harper, Westinghouse Electric Corp. , Aug. , 23, 1962.
- RI160 3-M Staff, "Scotchply Reinforced Plastics-Type 1100 High Strength Molding Compound", Minnesota Mining and Manufacturing Data Sheet #3, June 10, 1963.
- RI174 Staff, "Technical Data Report 61-22, Plaskon Alkyd Molding Compound 452", Allied Chemical Co. , 1963.
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- RI509 Brenner, W. ; Lum, W. ; Riley, M. , High Temperature Plastics, Reinhold, p. 54, 1962.

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- RI225 Traynor, E. S. ; Sampson, R. N. , Westinghouse Correspondence Feb. 4, 1964.
- RI240 Hysol Staff, "High Heat Distortion Hardener Hysol H5-3537", Technical Data E-217E, Hysol Corporation, December 1962.
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- RI253 Wechsler, K. W. , "Plastic Sheet Laminated Thermosetting Glass Cloth, MIL Grade GEB, Micarta H-2497", Westinghouse Physical Laboratory Report. Dec. 11, 1958.
- RI254 Staff Westinghouse, "Micarta Information Bulletin H-2497", Data Sheet, April 8, 1959.
- RI505 Stevens, G. H. , "Fatigue Test of Phenolic Laminate at High Stress Levels and Elevated Temperature", AD265 532 TAB, V. 62-1-2, p. 45, Jan. 15, 1962.
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## SECTION V

### BORE SEAL MATERIALS

#### A. INTRODUCTION.

The objective of this program is to identify properties of bore seal material systems which are capable of long life at 1000°F and 1600°F in potassium, sodium-potassium eutectic, and selectively to lithium.

The candidate materials for this program were selected on the basis of expected environmental compatibility, physical and mechanical properties and fabricability.

Static capsule tests will be used in the present program to determine alkali metal corrosion effects. High purity materials will be used in this study. The determination and control of the purity of alkali metals in the test capsule is being stressed, particularly in regard to oxygen contamination.

#### B. SUMMARY OF EFFORT THIS QUARTER.

The direction and status of the second quarter effort may be summarized as follows:

1. Capsule loading and environmental test facility planning are largely completed. Necessary equipment was designed and orders or fabrication initiated.
2. Material requirements were determined and orders initiated.
3. Test methods and associated jig and fixture requirements were ascertained.
4. Preliminary screening of ceramic-metal sealing systems was continued. Tensile and leak-test data on these systems were obtained.

## C. CAPSULE LOADING AND ENVIRONMENTAL TEST FACILITY.

### 1. Capsule Loading Facility

Details of a capsule loading facility and procedure which show promise of achieving minimum oxygen contamination in the loaded and sealed capsules have been determined. A schematic layout of the overall set-up is illustrated in Figure V-1.

The dry box is a General Technology Model Mark 5A modified to accommodate the special accessories for loading. A modified evacuation system is capable of an ultimate vacuum of less than  $1 \times 10^{-6}$  torr. It consists of a 15 cfm Welch forepump Model #1397B and a 400 liters/sec oil diffusion pump with a Freon 12 cooled chevron baffle to limit backstreaming. Associated vacuum manifolding permits pumping out the entry vestibule. The same pumping system is used to evacuate the loaded capsules prior to sealing. An ionization gage and power supply to monitor vacuum are included.

The argon atmosphere is supplied from a liquid argon reservoir and boiler capable of generating 50 cfm of gasified argon. The argon is passed through a General Dynamics Model HXV-200 Automatic Gas Purification System capable of purifying 50 cfm of the argon to less than 1 ppm of  $H_2O$  or  $O_2$ . Short connectors between the purification system and the dry box will be made of stainless steel.

Effluent argon from the dry box will be monitored for  $H_2O$  and  $O_2$  with a Beckman Hygromite and Model 80 Oxygen trace analyzer respectively. A dual track recorder will provide a permanent record of dry box conditions during capsule loading.

### 2. Capsule Loading Procedure

A sequence for capsule loading has been determined. The procedure below is for the loading of the ceramic modulus of rupture bars (0.1 inch x 0.1 inch x 1.0 inch) without braze joints. Some modifications will be dictated by experience.

- a. All required ceramic bars will be chemically cleaned, dried, weighed and stored in clean petri-dishes, and under dessicator conditions.
- b. Eight Cb-1% Zr sample capsules (0.5 inch O. C. x 0.049 inch wall x 4.0 inches long) with TIG welded bottom ends will be vacuum annealed ( $10^{-6}$  torr) at 2200°F for one hour. Capsule tops with tubulations will also be vacuum annealed. Parts will be stored in clean plastic bags.



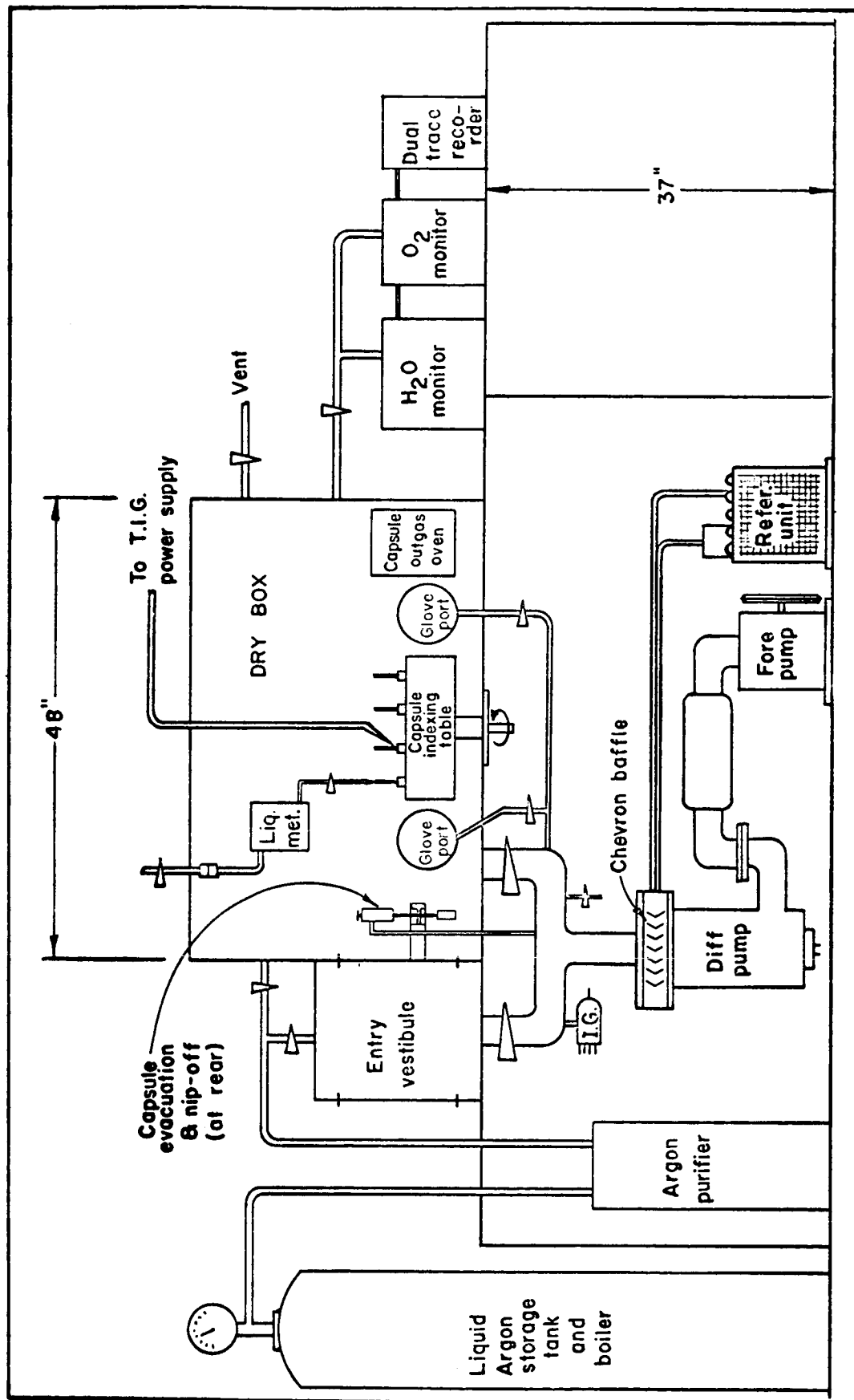


FIGURE V-1. Schematic of Capsule Loading Facility

- c. The alkali metal "purity test" capsule (0.375 inch O. D. x 0.065 inch wall x 4.0 inches long) with welded and tubulation top will be vacuum annealed.
- d. The selected alkali metal in its "hot trap" container will be hot trapped immediately before loading for a minimum of 24 hours at 1400°F.
- e. The glove box will be pumped to below  $10^{-4}$  torr and back filled with argon repeatedly until the O<sub>2</sub> and H<sub>2</sub>O levels remain relatively constant at less than 2 ppm combined.
- f. Items from a, b, c, and d above will be placed in the entry vestibule in clean metal racks (plastic bags and petri dish cover removed). The entry vestibule will then be evacuated and purged several times before opening the door into the main chamber.
- g. Specimens will be placed into the test capsules. The test pieces will be physically separated by jiggling and will rest on a platform well off the bottom of the capsule. The tubulation caps will then be put in place and TIG welded. Typical oxygen pickup in the weld area will have been determined previously on sample specimens by vacuum fusion analysis.
- h. The capsules, loaded with test assemblies, and with open tubulations will then be removed from the dry box and annealed for one hour at 2200°F in vacuum. This is possible if only ceramic materials are in the capsules. In the case of capsules containing brazed specimens, it will be necessary to keep the lower part of the capsule at less than the previous brazing temperature. For this reason, the bottom TIG welds will have been previously annealed.
- i. The annealed capsules will again be placed in the dry box by repeating steps e and f.
- j. The capsules will then be baked out in a small oven located in the glove box to remove gases which have been adsorbed on the surfaces during transfer between the vacuum anneal furnace and the dry box. This operation may be monitored by the analyzers on the glove box effluent gas.
- k. The capsules will then be loaded with a measured amount of alkali metal through the open tubulations by means of an extruder connected to the fitting on the hot trap alkali metal container. The extruder needle will be purged by extruding two needle volumes of alkali metal just prior to each capsule loading.

- l. Immediately after injecting with alkali metal, the capsules (one at a time) will be connected to the evacuation and nip-off system shown in Figure V-2.
- m. The capsules will be chilled to reduce the vapor pressure of the alkali metal to  $10^{-7}$  to  $10^{-8}$  torr and then evacuated to at least  $10^{-4}$  torr.
- n. The tubulation will then be sealed by nipping off with a hydraulic ram device as shown in Figure V-2.
- o. The nip-off will be TIG welded to insure a permanent seal.
- p. After loading, etc., all capsules including the purity test capsule, will be removed from the loading chamber and the tubulation TIG weld area will be vacuum annealed at 2200°F while the balance of the capsule and contents are kept below the subsequent exposure temperature (i. e. 1000°F max. ).
- q. The purity test capsule will be identified with the associated lot of capsules. The purity test capsule will be heated in vacuum for 1 hour at 1600°F to permit solution of capsule contaminants. The contents of the purity test capsule will then be analyzed for oxygen. The resulting analysis will be recorded and reported with the test data obtained on the associated test specimens.

### 3. Environmental Test Facility

The vacuum furnace which is to be used for heating the loaded capsules at the test temperatures of 1000°F and 1600°F is illustrated in Figure V-3 with additional furnace detail shown in Figure V-4. The pumping system, capable of ultimate pressures of less than  $10^{-6}$  torr, consists of a Welch 1397B, 15 cfm forepump, 400 liters/sec metal diffusion pump, a Freon 12 chilled chevron baffle, a constant level liquid nitrogen trap, and a manifold with ionization gage vacuum monitor and provisions for dry nitrogen letdown between cycles. The "potassium monitor" noted in Figure V-3 consists of two electrical terminals separated by 0.250 inch thick ceramic. In the event of a capsule leak, the released vapor will condense out on the ceramic and lower the resistance between the terminals. The interterminal resistance will be constantly monitored with a simple bridge circuit and recorder.

The furnace temperatures will be monitored with thermocouples attached to both top and bottom of a representative capsule. This unit is under construction.

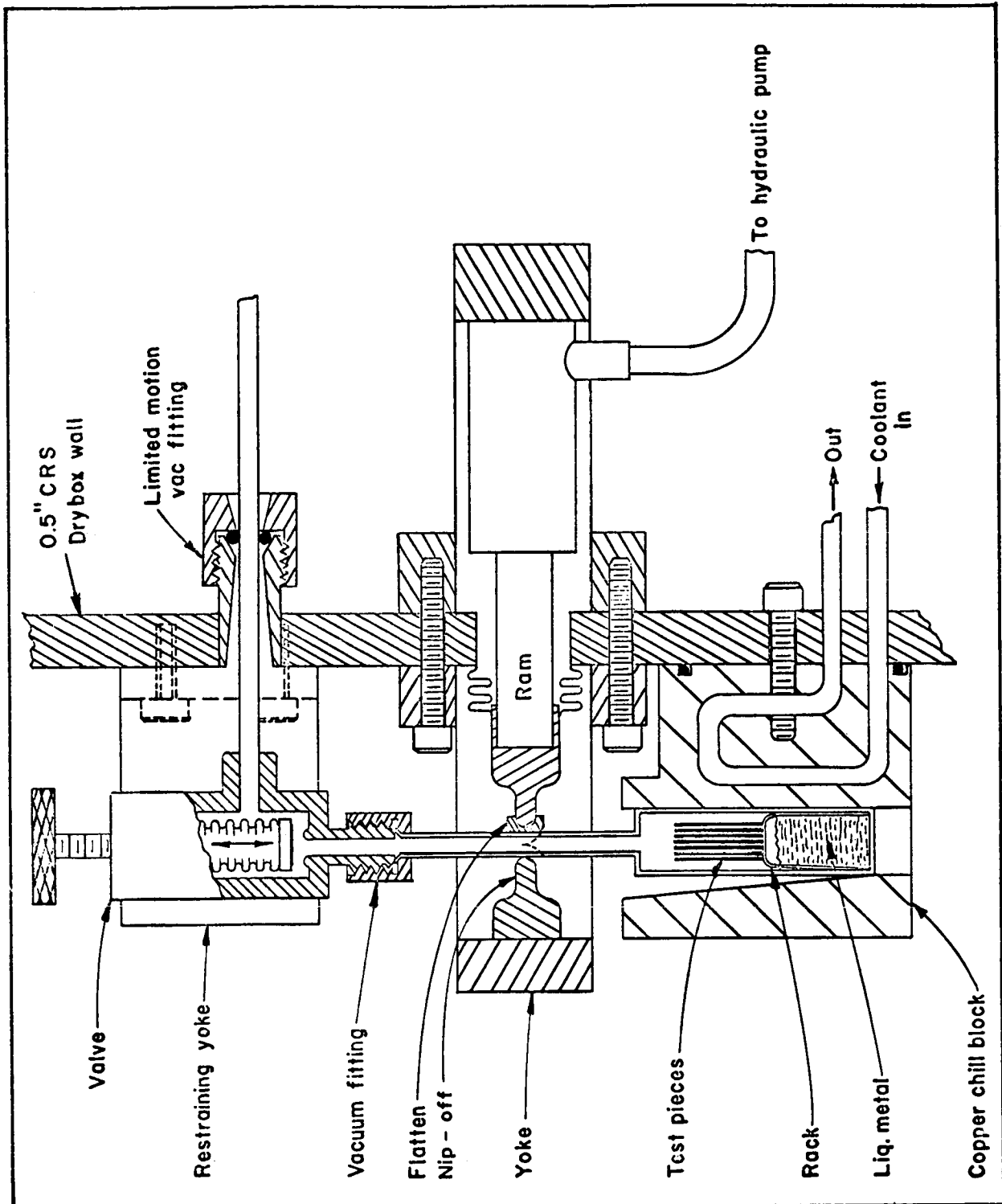


FIGURE V-2. Schematic of Capsule Evacuation And Nip-Off System

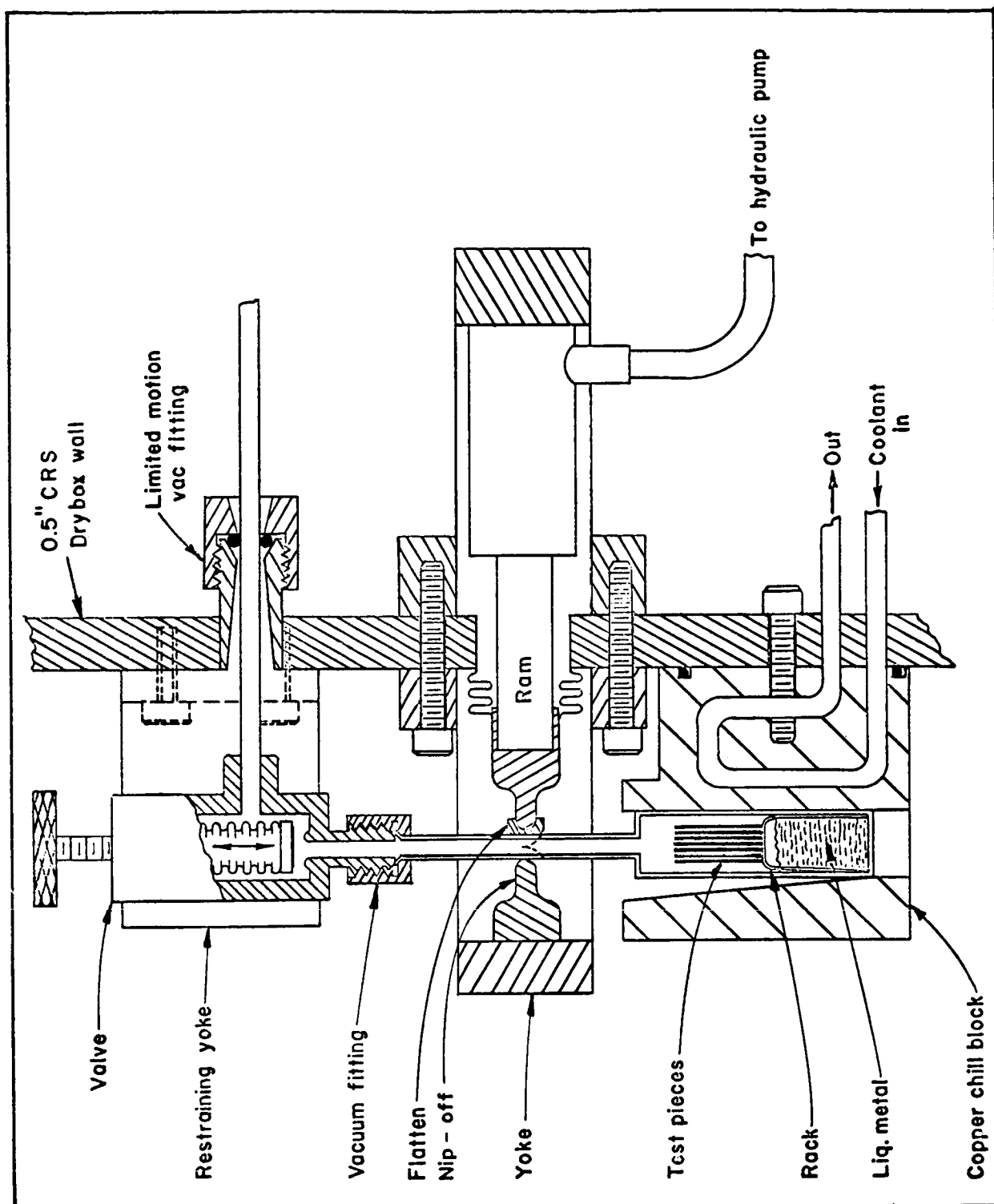


FIGURE V-2. Schematic of Capsule Evacuation And Nip-Off System

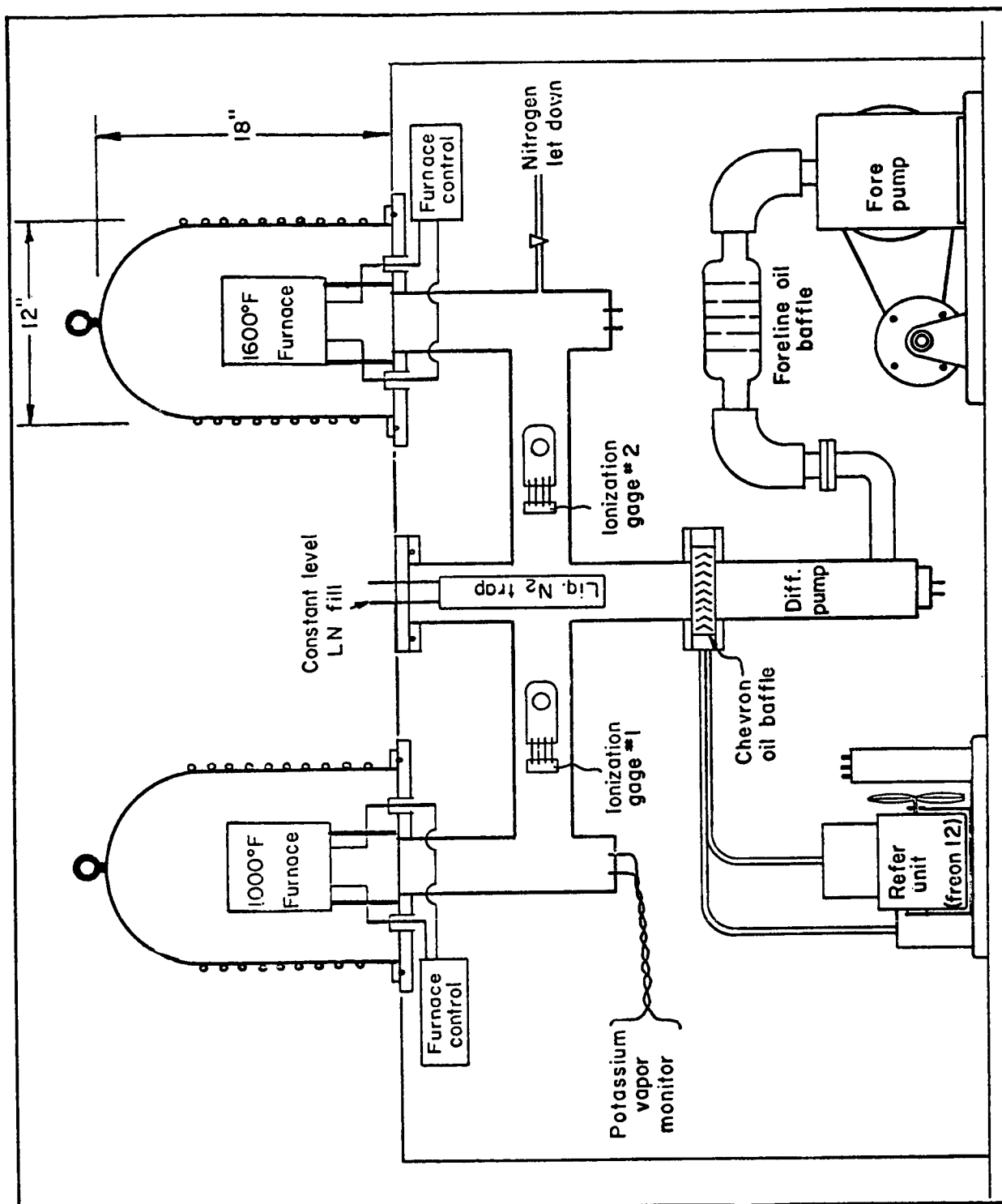


FIGURE V-3. Schematic of Environmental Test Facility

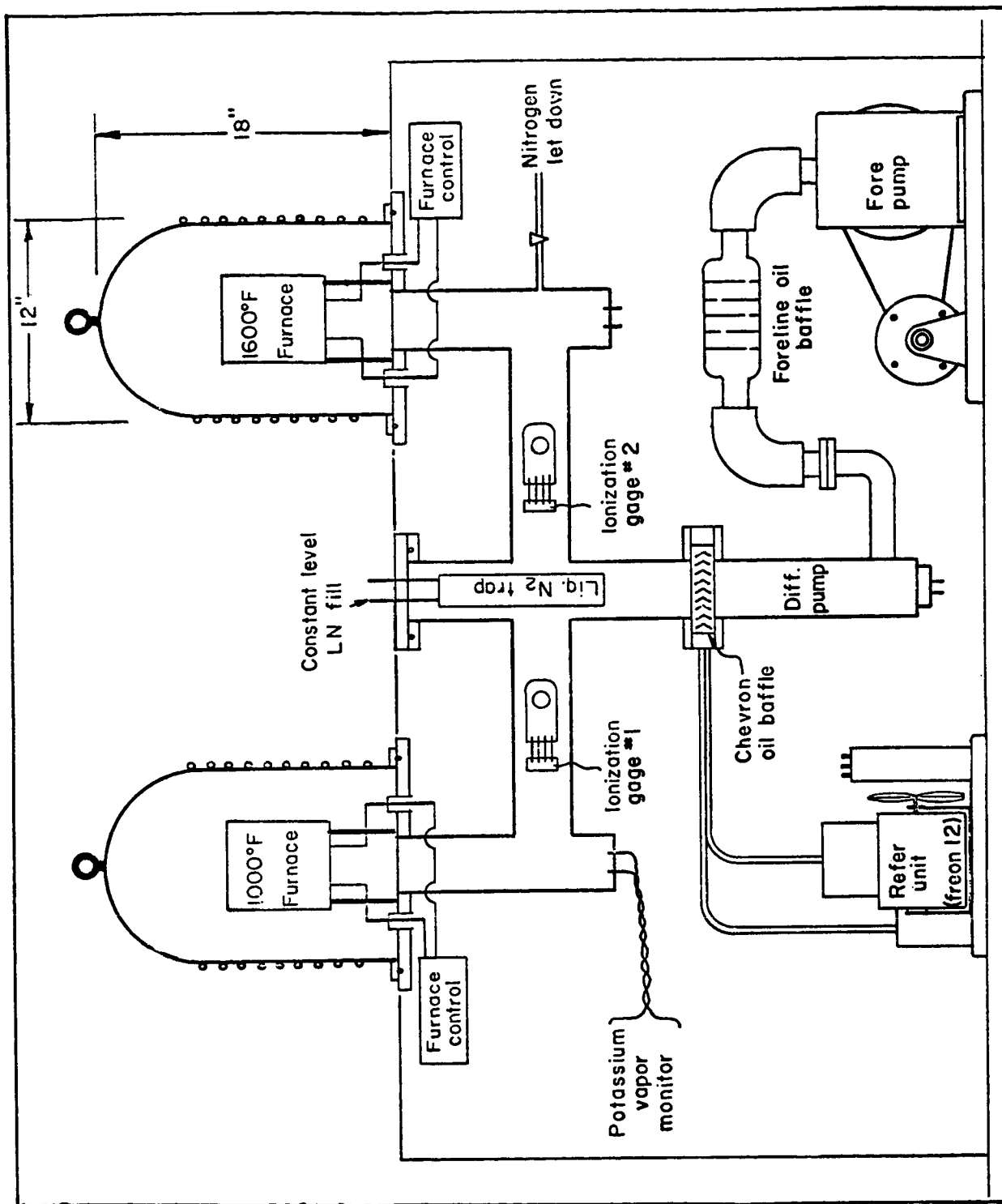


FIGURE V-3. Schematic of Environmental Test Facility

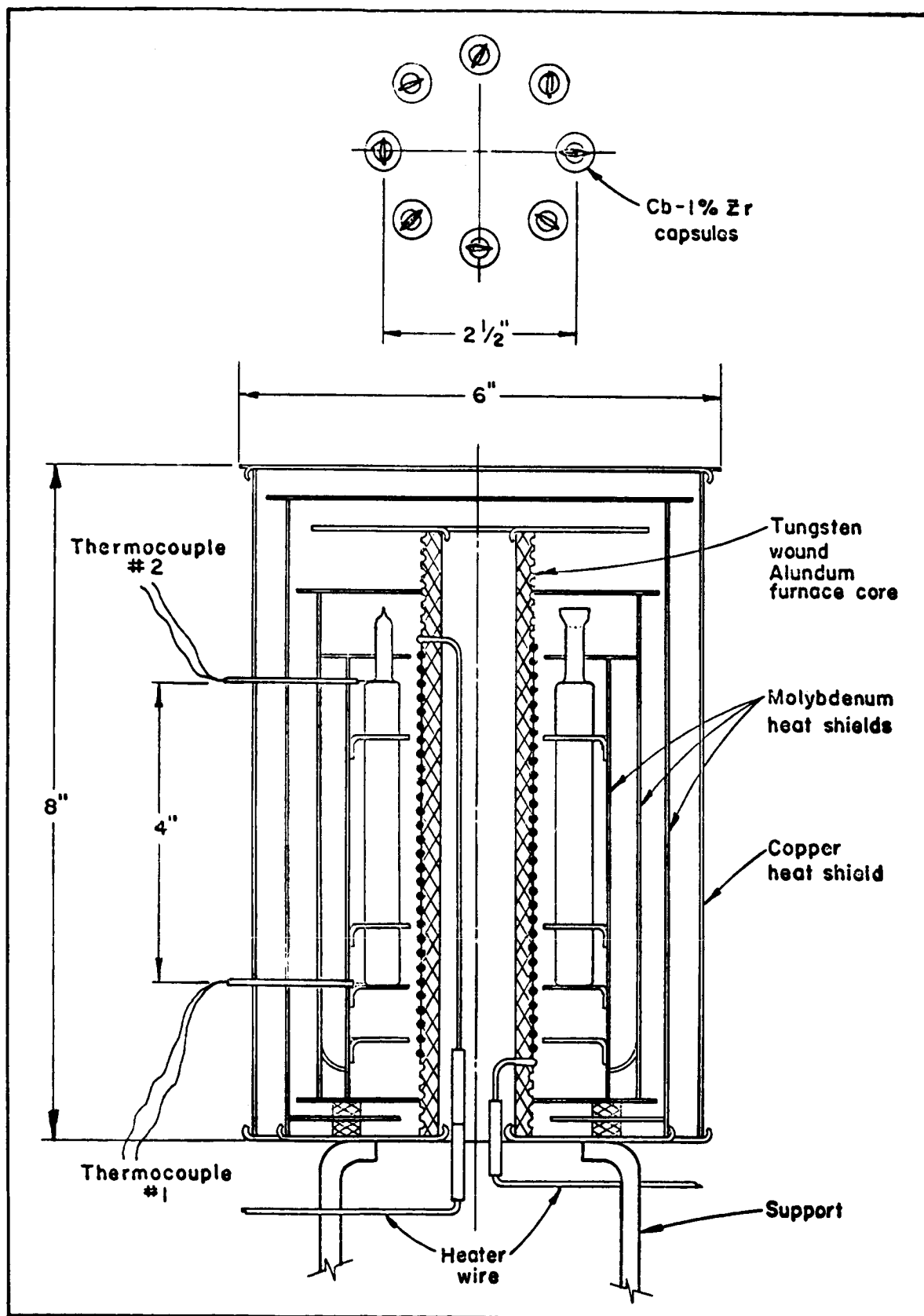


FIGURE V-4. Schematic of Furnace Detail

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#### 4. Environmental Test Procedure

The sealed, loaded capsules will be wrapped with Ta and Zr foil to limit oxygen pickup by the Cb-1% Zr capsule and then placed in the vacuum furnace. The system will then be evacuated and the furnace temperature raised slowly enough that the vacuum does not exceed the  $10^{-6}$  torr scale. The ultimate pressure is  $1 \times 10^{-6}$  torr.

The test duration on this program is 500 hours at temperature. During this period the liquid nitrogen trap will be kept automatically at a constant level, and the potassium vapor detector will be operated continuously.

The furnace temperature will then be reduced to room temperature, the system let down to dry nitrogen, and the capsules removed.

## D. MATERIAL STUDIES.

### 1. Ceramics

Based on the information presented in the first quarterly report, ceramics with the best alkali metal containment possibilities have been selected for evaluation as bore seal materials. The ceramic materials were placed in the following groups:

#### a) Material Requirements

The ceramic materials to be evaluated were selected from the following categories:

Group I	99+% $\text{Al}_2\text{O}_3$
Group II	98+% $\text{Al}_2\text{O}_3$
Group III	99+% $\text{BeO}$
Group IV	Special Composition

The typical composition of selected materials are given in Table V-1. All five bodies have been ordered in the form of 30 (M-of-R) modulus of rupture bars.  $\text{Y}_2\text{O}_3$  and  $\text{Dy}_2\text{O}_3$  have been ordered for the group IV bodies in 1/2 and 1/4 pound lots respectively. To date the chemical analyses of the Coors, Linde and Wesgo bodies have not been received. When all bodies have been received, they will be subjected to spectrographic analysis as a group to permit a direct comparison of trace element content including silica.

### 2. Metal Member

With the few exceptions noted in the first quarterly report, the physical and mechanical properties of the metals being considered for bore seal members are adequately documented. The effort on this program will be directed toward determining the joining characteristics of the metals in the ceramic to metal system and the determination of the alkali metal corrosion resistance of the systems. Sample seals will be fabricated and evaluated using inserts of columbium-1% zirconium alloy, columbium D-43 alloy and tantalum T-111 alloy. Columbium-1% zirconium will be used as the capsule material in this program.

The Cb-1% Zr alloy is not available in the desired tubing sizes (0.70 inch and 0.30 inch O. D. with 0.1 inch wall) without excessive tooling charges. The 0.1 inch wall was selected for its ability to contain lithium at 1800°F

TABLE V-1. Nominal Composition of Selected Ceramics

Group	Body	% Major Constituent	Flux Phase Composition (%)			Other
			MgO	CaO	SiO <sub>2</sub>	
I	Linde Sapphire	100% Al <sub>2</sub> O <sub>3</sub>	---	---	---	---
I	Lucalox (G. E. )	99. 75% Al <sub>2</sub> O <sub>3</sub>	0. 25	---	---	---
II	Ei-3-3W (Wesgo)	99. 7+ % Al <sub>2</sub> O <sub>3</sub>	0. 1	0. 1	<0. 1	<0. 01
II	AD-99 (Coors)	99% Al <sub>2</sub> O <sub>3</sub>	0. 25	0. 25	0. 5	<0. 1
III	F-1 (Brush)	99. 8% BeO	0. 130*	0. 0085*	0. 008*	Al 0. 015* Fe 0. 006*
IV	Special	**	**	**	**	**
<p>*Lot analyses on supplied material. All other elements present at less than 0. 003% (30 ppm).</p> <p>**To be fabricated from CaO, MgO or the rare earths.</p>						

for 1000 hours without leaking<sup>1</sup>. The less severe requirement of 500 hours at 1600°F may be met with 0. 050 inch material. This thinner material is on order in the form of 0. 5 inch, 0. 375 inch, and 0. 25 inch (tubulation) O. D. tubing and 0. 050 inch sheet. Zirconium and tantalum foil (0. 002 inch thick) and 0. 015 inch thick Cb-1% Zr have been received.

<sup>1</sup>D. Elliot, Private Communication, Jet Propulsion Laboratory, Pasadena, California.

## **E. TEST METHODS.**

The purity of all materials used will be determined by the suppliers lot analysis and by independent analyses where necessary and possible. Fabrication history will also be supplied.

The ceramics and ceramic-metal seals will be evaluated mechanically and metallurgically by means of six basic geometries.

- 1) ASTM CLM-15 Tensile test piece (Figure V-5)
- 2) Modulus-of-Rupture (M-of-R) bar (0.1 inch x 0.1 inch x 1.0 inch (Figure V-6a)
- 3) M-of-R rod (0.1 inch diameter x 1.0 inch long) (Figure V-6b)
- 4) M-of-R assembly (Figure V-6c)
- 5) Tab Peel assembly (Figure V-6d)
- 6) Vacuum test assembly (Figure V-6e)

The M-of-R brazed assemblies will be made up with oversize bars (0.110 inch) and ground back to noted dimensions (0.100 inch) before testing. This will eliminate the effects of braze fillets. The brazing jig is shown in Figure V-7. The tab peel test assemblies will be ground as noted in Figure V-6d for the same reason.

Four point loading will be used for obtaining strength data on the M-of-R bars, rods and assemblies, and will utilize the fixture shown in Figure V-8 and the formulas in Table V-2. This fixture is nearing completion. Tensile strength and modulus of rupture correlation between the ASTM CLM 15 assembly and the M-of-R assembly with identical sealing systems will be determined. Drum peel data using the O. D. of the CLM 15 test piece and tab peel data will also be compared. Test fixtures for these tests are shown in Figure V-9. The square and round M-of-R test piece strengths must also be correlated. The latter comparison is required because of the extremely high cost of the square M-of-R geometry in sapphire.

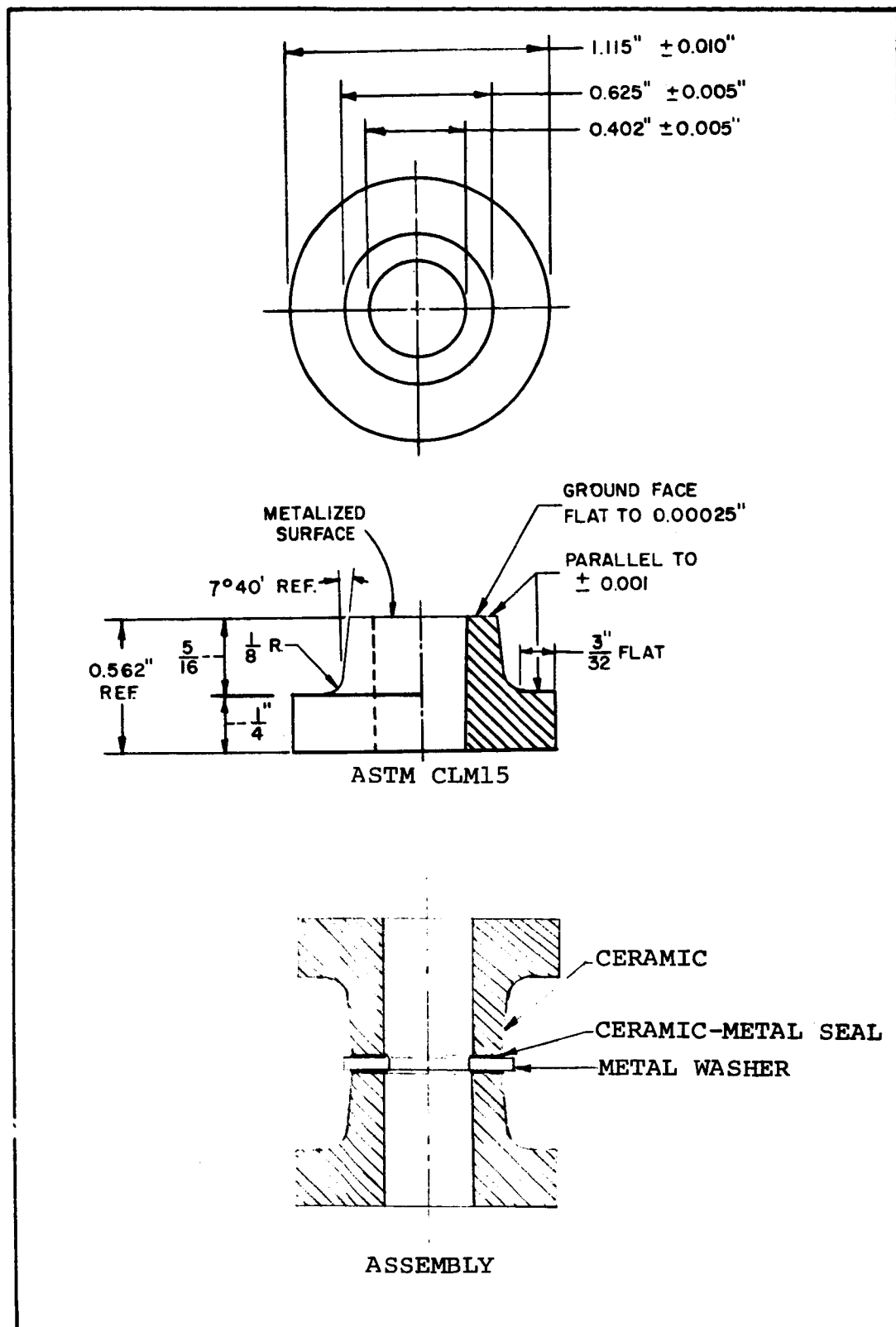


FIGURE V-5. Tensile Test Piece ASTM CLM15  
And Assembly

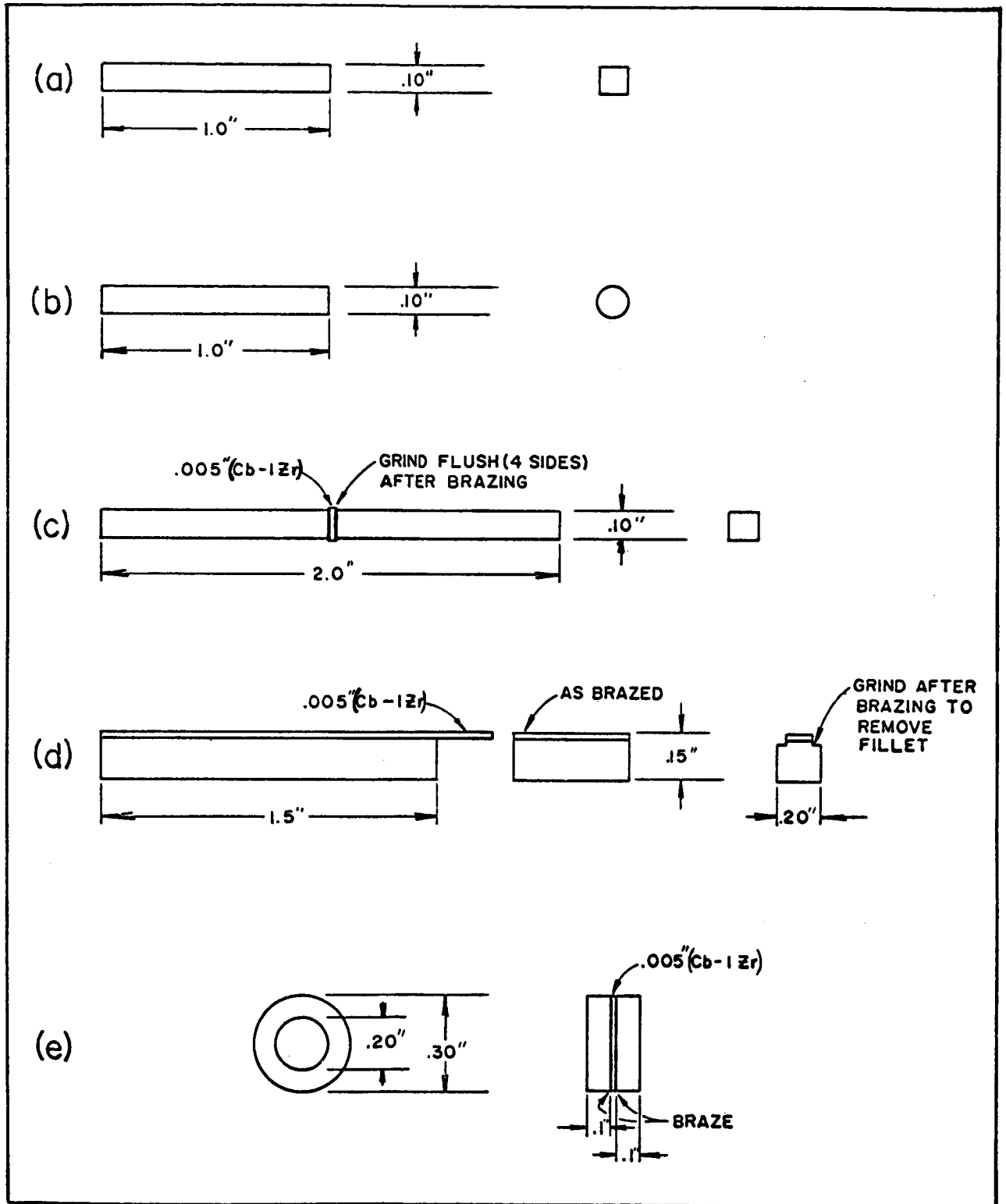


FIGURE V-6. Test Assembly Geometries

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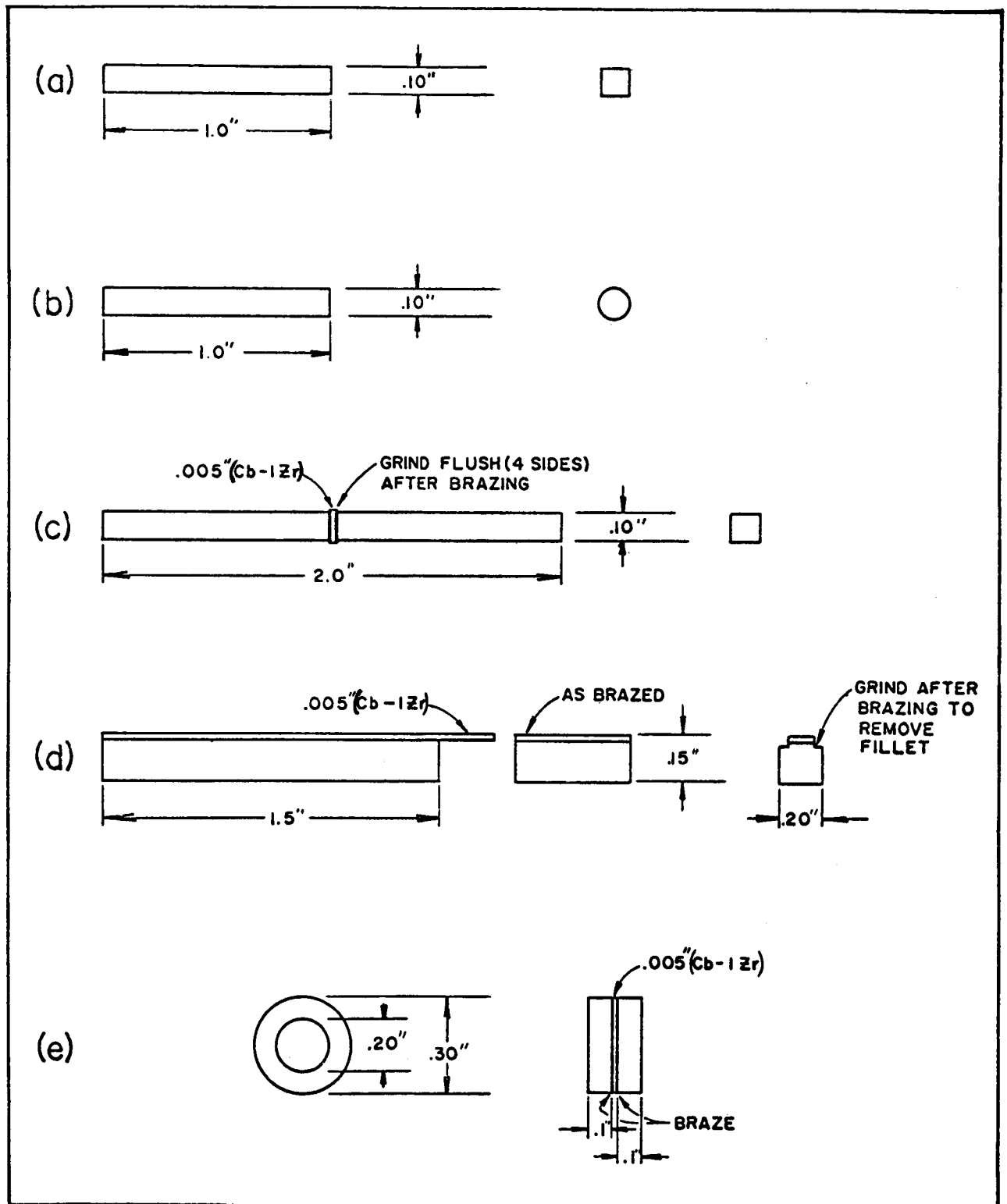


FIGURE V-6. Test Assembly Geometries

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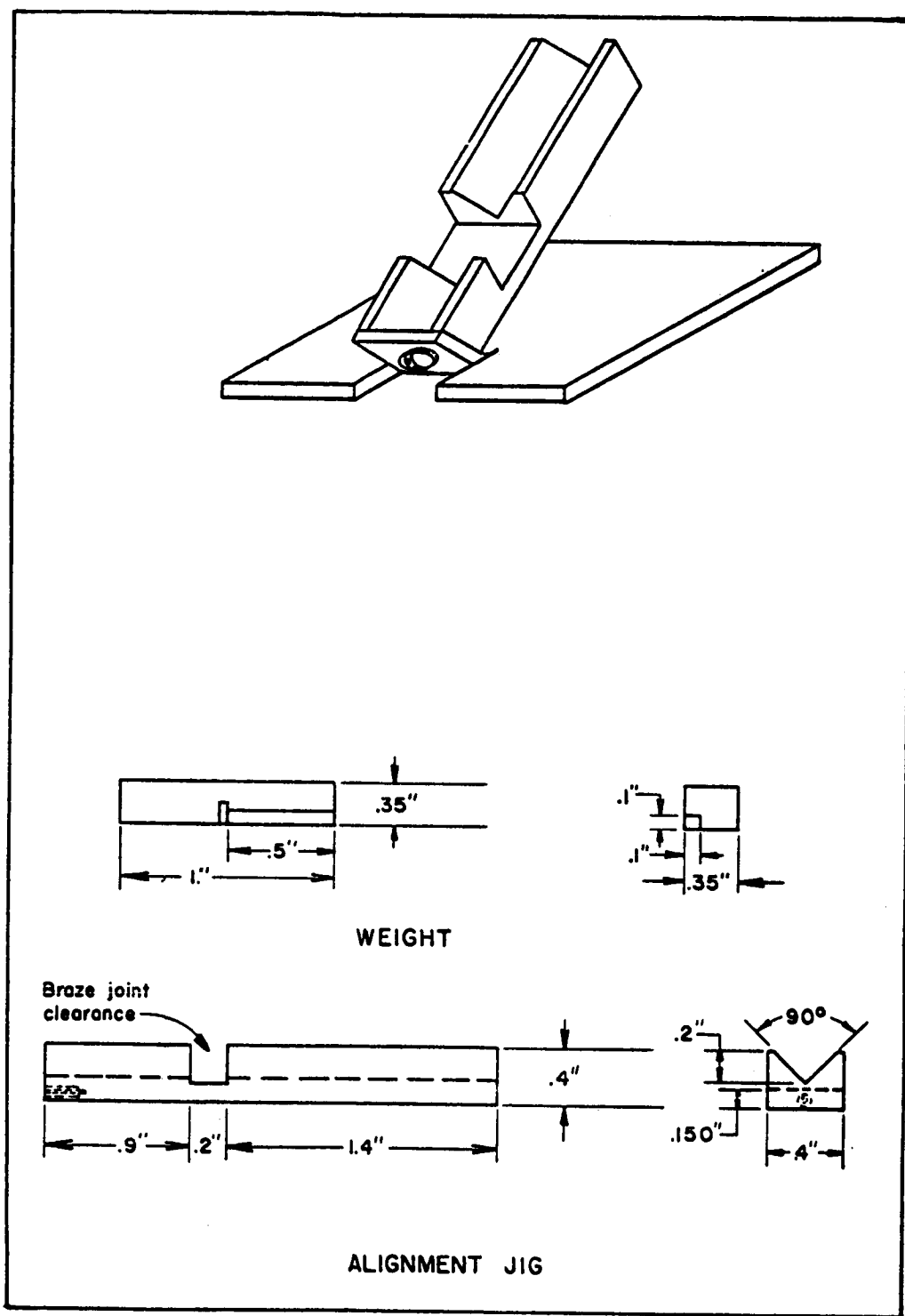


FIGURE V-7. Modulus of Rupture Brazing Fixture



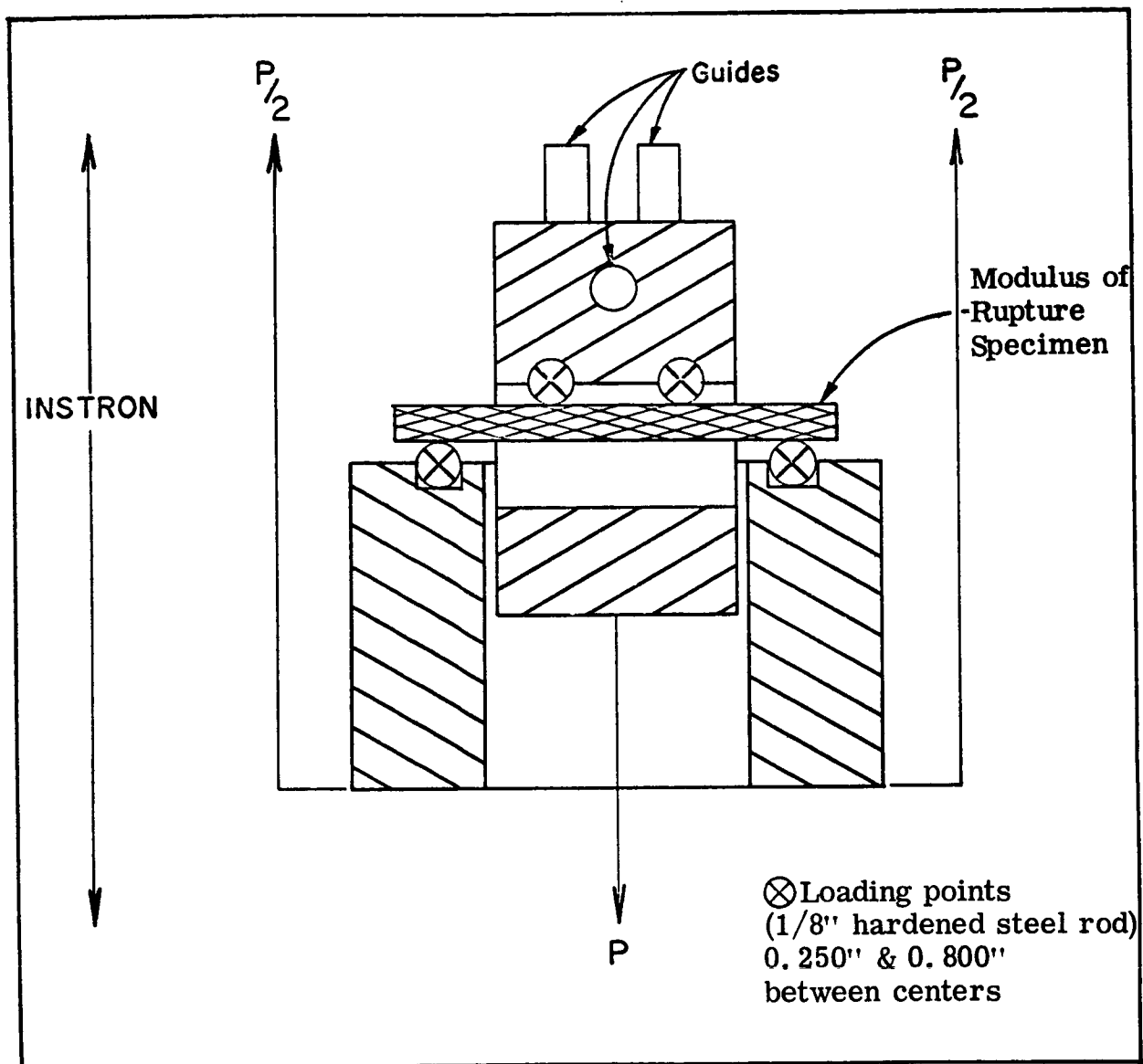
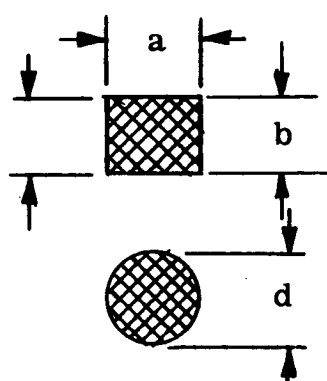
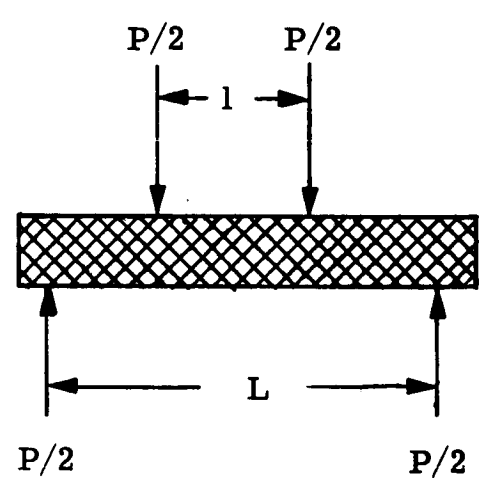


FIGURE V-8. Four Point Loading Fixture

TABLE V-2. Formulas For Computing Modulus of Rupture  
(Flexural Strength)

Cross Section	Formula
	$M \text{ of } R = \frac{3(L-1)P}{2ab^2}$ $M \text{ of } R = \frac{8(L-1)P}{\pi d^3}$

Symbol	Units
M of R	psi
a, b, d	inches
P	pounds
L, l	inches



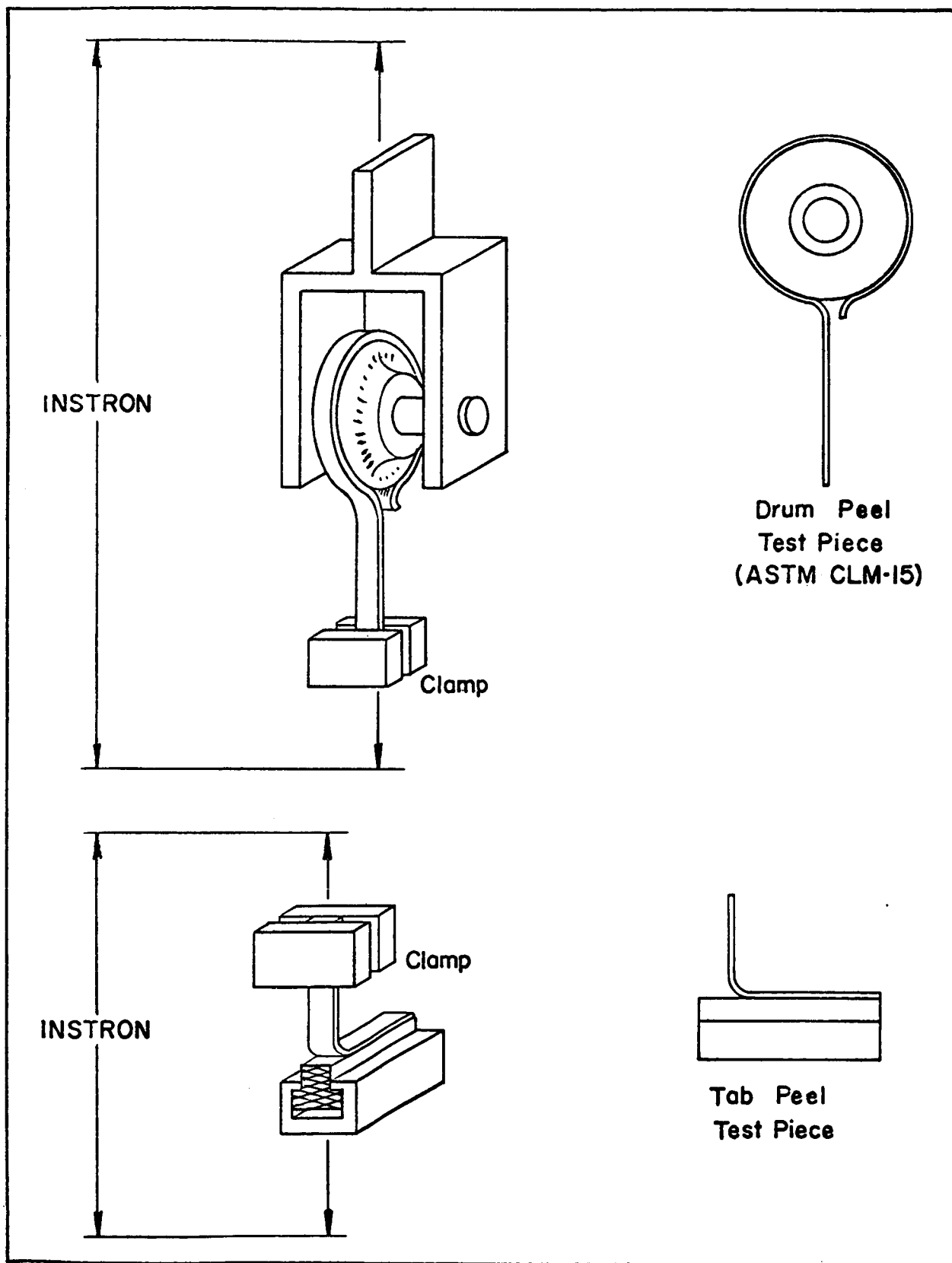


FIGURE V-9. Drum And Tab Peel Test Fixtures

WAED64. 14E-127

## F. CERAMIC TO METAL SEALING SYSTEMS.

The sealing system remains the most critical area in current bore-seal technology. The two methods which are usually used to seal ceramics to metals are the refractory metal metalizing braze system and the active metal braze process.

### 1. Metalizing - Braze System

Conventional refractory metal metalizing compositions contain, in addition to the molybdenum or tungsten metal, a non-metallic phase consisting of materials such as  $\text{MnO}$ ,  $\text{TiO}_2$ ,  $\text{SiO}_2$ , or combinations of these. Previous related work<sup>2</sup> indicates that a molybdenum base paint containing  $\text{MnO-TiO}_2$  as the non-metallic phase is attacked by potassium vapor at  $1100^\circ\text{F}$ .

Based on thermodynamic stability criteria, the following partial list of metalizings are potentially useful in potassium and NaK vapors.

- a. Pure Mo or  $\text{MoO}_3$
- b. Pure W or  $\text{WO}_3$
- c. W-CaO- $\text{Al}_2\text{O}_3$
- d. W-CaO-MgO- $\text{Al}_2\text{O}_3$
- e. Mo- $\text{Y}_2\text{O}_3$
- f. W- $\text{Y}_2\text{O}_3$
- g. Mixtures of the above
- h. Other rare earth oxide substitutions.

None of the normal refractory metal metalizings are expected to be suitable in lithium. However, evaporated coatings or cermet type transition layers of columbium, tungsten, molybdenum, or rhenium offer promise. A limited effort in applying the cermet transition joining method is scheduled on this program.

To metalize large ceramic bodies, the firing temperature should be in the  $3000^\circ\text{F}$  range. The tungsten-yttria composition has been successfully metalized on both Coors AD995 body ( $\text{Al}_2\text{O}_3$ , 0.2%,  $\text{SiO}_2$ , 0.5% Mg, 0.2% Cr) and

<sup>2</sup> Westinghouse Subcontract on SPUR Project, AF33(657)10922.

on Lucalox at 3000°F to give high strength bonds.<sup>3</sup> This is a development suggested by the work of Stoddard and Cowan (LB145). \*

The phase diagram of  $Y_2O_3 - Al_2O_3$  indicates that the eutectic melting point is 3200°F.<sup>4</sup> It is probable, therefore, that successful metalizing depends on the MgO and/or  $SiO_2$  in the bodies noted above to reduce the metalizing temperature. A further reduction in temperature could be achieved by adding other stable oxides to the metalizing paint.

Coast Metals Alloy 52 and pure copper have indicated stability in potassium vapor to 1100°F for 1000 hours.<sup>5</sup> The former will be screened for possible use in potassium and NaK at higher temperatures. An iron overplate on the metalizing is used with Alloy 52.

With these considerations in mind, two series of tungsten base metalizing paints were compounded with rare earth and other oxides as additives. The first series of paints was made containing five percent of an alkali metal compatible non-metallic phase. Subsequently a group of paints was made with fifteen percent non-metallic phase added to reduce the observed porosity on specimens made with the five percent paints. Paint compositions are given in Table V-3. Typical spectrographic analyses of trace impurities in the first group of paints are given in Table V-4. Nominal compositions of the substrate materials are given in Table V-1.

The metalizing paints were made in molybdenum lined ball mills containing molybdenum grinding balls to minimize pick-up of non-metallic phase during the paint milling operation.

Paints are evaluated by brazing ASTM CLM 15 assemblies with copper which does not react with the metalizing. The metalizing-to-ceramic bond is thus measured under optimum conditions. Paints which exhibit satisfactory strengths are then brazed with potentially useful braze alloys such as Coast Metals Alloy 52 (3% B, 4.5% Si, 0.15% C, Ni balance) and with a 0.015 inch Cb washer between the two halves of the CLM 15 assembly. Both the tungsten metalizing and Cb washers are plated with 0.0005 inch of Fe and sintered in vacuum for 10 minutes at 1475°F. This plated layer limits reaction between

\*LB References noted are listed at the end of this section.

<sup>3</sup>C. Ritz, Private Communication, Bendix Corp., Red Bank, N. J.

<sup>4</sup>I. Warshaw and R. Roy, "Stable and Meta-stable Equilibria in the Systems  $Y_2O_3-Al_2O_3$  and  $Gd_2O_3-Fe_2O_3$ ," J. Amer. Ceramic Soc. V. 42, P. 435, 1959.

<sup>5</sup>Westinghouse Subcontract SPUR Project AF33(657)8954, and Westinghouse Subcontract AF33(657)10922, Westinghouse Report No. WAED62.2E of January, 1964.

**TABLE V-3. Metalizing Paints for Alkali Metal Environments  
(For Reference Only)**

Paint ** Code	Nominal Composition (Wt. %)	
	Metallic Phase	Non-metallic Phase
W5M	95% W*	5%Y <sub>2</sub> O <sub>3</sub>
W8M	95% W*	2% Al <sub>2</sub> O <sub>3</sub> , 3% CaCO <sub>3</sub>
W9M	95% W*	2.5%Y <sub>2</sub> O <sub>3</sub> , 2.5% Dy <sub>2</sub> O <sub>3</sub>
W10M	95% W*	1.25%Y <sub>2</sub> O <sub>3</sub> , 1.25% Dy <sub>2</sub> O <sub>3</sub> 1.25% Nd <sub>2</sub> O <sub>3</sub> , 1.25% Gd <sub>2</sub> O <sub>3</sub>
W11M	85% W*	15%Y <sub>2</sub> O <sub>3</sub>
W12M	85% W*	6%Al <sub>2</sub> O <sub>3</sub> , 9% CaCO <sub>3</sub>
W13M	85% W*	3.75%Y <sub>2</sub> O <sub>3</sub> , 3.75% Dy <sub>2</sub> O <sub>3</sub> 3.75% Nd <sub>2</sub> O <sub>3</sub> , 3.75% Gd <sub>2</sub> O <sub>3</sub>
W14M	95% W*	2.5%Y <sub>2</sub> O <sub>3</sub> , 1% Al <sub>2</sub> O <sub>3</sub> , 1.5% CaCO <sub>3</sub>
W15M	92% W*	5.5% Y <sub>2</sub> O <sub>3</sub> , .5% MgO 2% CaCO <sub>3</sub>
<p>*Added as WO<sub>3</sub>.</p> <p>**Eitel - McCullough Designation</p>		

TABLE V-4. Spectrographic Analyses of Trace Impurities in  
Special Metalizing Paints (Typical Analysis)

Paint*** Code	Impurity (Wt %)*							
	Mg	Al	Cu	Ca	Si	Mn	Fe	Mo
W5M	T**	0.007	T	0.01	0.02	ND	0.02	0.02
W8M	0.0015	PC	T	PC	0.025	T	0.02	0.02
W9M	T	0.006	0.001	0.02	0.015	T	0.02	ND
W10M	T	0.003	T	0.01	0.01	T	0.01	ND
PC - Primary Constituent ND - Not Determined *Semi-quantitative only $\pm$ factor of 4. **T indicates less than 0.001 wt % detected. ***Eitel - McCullough Designation								

the nickel alloy braze, the metalizing and the columbium washer. The special CLM 15 assemblies are normally brazed to a production metalized 94% alumina (AD94) CLM 15 piece as a mating member for expediency. This does not interfere with the validity of the test.

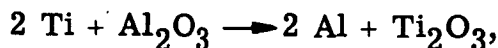
Table V-5 lists the tensile and vacuum test data obtained to date on these assemblies. Limited test data have been obtained on the nickel alloy brazed tungsten base paints. Two paints, W12M and W13M, have produced vacuum tight seals to columbium with nickel alloy brazes.

The W12M, W14M, and W15M paints applied to Ei 3-3 bodies, exhibited low tensile strengths. Upon examination of the specimens, it was determined that the break occurred in the seal on the AD94 side of the test specimen. All other specimens broke in the ceramic indicating a metalizing strength greater than that shown in Table V-5.

Polished cross sections of copper brazed, tungsten metalized (5% non-metallic phase) pieces were made. Metallographic examination revealed infiltration of copper into the metalizing. The indication of porosity led to the blending of the 15% non-metallic paints. Seal evaluation is not complete at this time. Peel test results and photomicrographs will appear in the next report.

## 2. Active Metal Brazing

An active metal braze joint can be made directly between ceramic and metal. Titanium active metal brazes resist potassium vapor but the possibility of the reaction



provides an interface layer of  $\text{Ti}_2\text{O}_3$ , a compound of moderate stability in NaK, K, or Li. In one observation, microprobe determination of aluminum in the braze alloy indicated a maximum pickup of 0.2% Al in the braze alloy.<sup>6</sup> The use of refractory metals in active metal brazing in conjunction with a metalized surface circumvents this difficulty.

The first criterion for the use of active metal alloy is that it be ductile and that its thermal expansion matches that of the ceramic as closely as possible so that the joint will not crack and leak in service. A second criterion is that the active alloy be compatible with the columbium member and with the alkali metal vapor environment. A brazing temperature below 2000°F is desired for ease of fabrication.

<sup>6</sup>R. H. Bristow, et al, of G. E. , Report R572F "Final Technical Summary Report on Vapor Filled Thermionic Converter and Joining Problems", Contract Nobs 86220 Project No. Sr 007-1201, Feb. 1963.



TABLE V-5. Tensile Strength and Leak Testing of Special Metalizing Paints  
Utilizing ASTM, CLM 15 Tensile Test Assembly

Paint Symbol	Copper Braze (a)						Nickel Alloy Braze (b)		
	Ei-3-3 (99.79% Al <sub>2</sub> O <sub>3</sub> )			AD89 (99% Al <sub>2</sub> O <sub>3</sub> )			AD89 (99% Al <sub>2</sub> O <sub>3</sub> )		
	No. of Tests	Tensile Strength	No. of Tests	Leak Test	No. of Tests	Tensile Strength	No. of Tests	Tensile Strength	Leak Test
W5M	1	>14,850 psi	2	VT					
W8M	1	>12,900 psi	2	VT					
W9M	1	>14,550 psi	2	VT					
W10M	1	>13,200 psi	2	VT					
W11M	1	>14,400 psi	1	VT	2	>12,600 psi	2	--	1 LKR
W12M	1	6,030 (c) psi	1	LKR	2	>11,250 psi	2	--	1 VT
W13M	1	>12,900 psi	1	VT	2	>11,160 psi	2	--	1 VT
W14M	1	8,160 (c) psi	1	VT	2	>13,110 psi	2	--	1 LKR
W15M	1	4,050 (c) psi	1	VT	2	>13,290 psi	2	--	1 LKR

The prefix (>) indicates a metalizing strength greater than the figure shown. The specimen broke in the ceramic at the stress level noted.

VT indicates a leak rate less than  $1 \times 10^{-9}$  torr-liters/sec as determined in leak testing procedure in Appendix A. LKR indicates leaker.

All paints listed were sintered for 1/2 hour at 3045°F in forming gas, 75% N<sub>2</sub>, 25% H<sub>2</sub>, 70°F dewpoint.

- (a) Copper brazed in -100°F dewpoint hydrogen at 2040°F with 3 minute hold at temperature. 0.020" 70/30 cupro-nickel washer between CLM-15 pieces.
- (b) Braze Alloy 52 brazed at 1850°F in vacuum with no hold time at temperature. 0.015" Cb washer between CLM-15 pieces. Cb washer and metalizing plated with 0.0005" of Fe; vacuum sintered for 10 min. at 1470°F.
- (c) Specimens broke in the AD94 side of the joint.

The work at the Oak Ridge National Laboratory on brazing materials for columbium (LB20) has resulted in alloys which appear promising for active-metal brazing. These alloys, listed in Table V-6, have been given preliminary screening on a related contract brazing  $\text{Al}_2\text{O}_3$ .<sup>7</sup> An additional alloy AS-537 (LB24) was evaluated on the latter program.

Work is under way on this program (NAS3-4162) to extend the screening evaluation of the promising alloys for brazing to BeO ceramics. Preliminary testing has started using 98% BeO ceramics. Hermetic seals have been obtained with the following brazing alloys:

- a. 48 Zr - 48 Ti - 4 Be
- b. 46 Ti - 46 Zr - 4 V - Be
- c. 56 Zr - 28 V - 16 Ti

<sup>7</sup>Westinghouse Subcontract SPUR Project AF33(657)8954, and Westinghouse Subcontract AF33(657)10922, Westinghouse Report No. WAED64.2E of January, 1964.

TABLE V-6. Experimental Alloys for Brazing Columbium

No.	Ref. No.	Alloy (Wt. %)	Flow Point (°F)
1	LB20	67Zr-29V-4Fe	2370
2	LB20	60Zr-25V-15Cb	2340
3	LB20	48Zr-48Ti-4Be	1920
4	LB20	63Ti-27Fe-10Mo	2280
5	LB20	63Ti-27Fe-10V	2340
6	LB20	68Ti-28V-4Be	2280
7	LB20	45Ti-40Zr-15Fe	1920
8	LB20	75Zr-19Cb-6Be	1920
9	LB20	46Ti-46Zr-4V-4Be	1830
10	LB20	95Zr-5Be	1830
11	LB20	62Ti-26Fe-8Mo-4Zr	2280
12	LB20	80Zr-17Fe-3Be	1830
13	LB24	56Zr-28V-16Ti (AS-537)	2280

## G. THIN FILM METALIZING.

Evaporated thin film refractory metal metalizing was previously developed at Eitel-McCullough\* and appears promising for bore seal applications. Tensile strengths of over 15,000 psi are obtainable without a non-metallic phase in the sealing system. Vacuum-tight seals to BeO have been fabricated by this method. Preliminary work was done with evaporated molybdenum and copper brazing. Nickel alloy brazing is being evaluated with Fe and Cr "barrier layers" over the thin metalizing to prevent erosion during the brazing operation.

A summary of both copper and nickel alloy brazing results on this program is presented in Table V-7. A seal, which had been fabricated with a Coors AD99 (99%  $\text{Al}_2\text{O}_3$ ) ASTM CLM 15 tensile specimen using thin film metalizing techniques, was sectioned for metallurgical examination. The seal was made by evaporating a film of titanium less than one micron thick on the ceramic body followed by a layer of molybdenum slightly greater than one micron thick. After a 0.0005 inch thick plating of iron was applied, the part was sintered at 1475°F for ten minutes in vacuum. The prepared ceramic was then brazed in a vacuum of  $10^{-6}$  torr at 1850°F to iron plated 0.015 inch thick columbium with braze alloy 52 (3% B, 4.5% Si, 0.15% C, bal. Ni). The other half of the CLM 15 assembly consisted of an AD94 ceramic, metalized with Mo-MnO-TiO and iron plated as above. The seal indicated a leak under vacuum and had a tensile strength of 6,400 psi. In Figure V-10 a photomicrograph of the thin film metalized side of the seal is shown. The iron plating is intact on both the columbium and metalized ceramic. The possibility of ruptures in the iron plate and erosion of the thin molybdenum layer by the brazing alloy in other portions of the seal exists, with associated degradation of the metalizing-ceramic bond. The iron plate may also be reacting with the thin molybdenum with similar degradation. The molybdenum layer is seen to have separated from the ceramic in the photomicrograph. If this did not occur in the sample preparation, it is significant and indicates that the evaporated molybdenum metalizing is not suitable for use in a highly stressed seal. This does not occur with copper brazes which are more ductile than Alloy 52. A copper-brazed seal is shown in Figure V-11.

Additional work with polished and unpolished ceramic surfaces and with chrome substituted for the iron barrier layer are planned to resolve the failure mechanism. Electron microprobe analyses across the molybdenum layer will be required to determine the extent of iron diffusion.

\*Patent No. 3,115,957 - other pending.

TABLE V-7. Tensile Strength and Leak Testing of Thin Film  
Metalizing on CLM-15 Assemblies

Ceramic	Metalizing	Plate	Vacuum Braze	Metal Member	Tensile Strength	Leak (a) Test
AD995 (pol)	(b)	Cu flash	Cu	Cupro Nickel	>15, 500 psi	(1) VT
AD995 (pol)	(b)	Cu flash	Cu	Cupro Nickel	>13, 375 psi	(1) VT
AD995	(b)	Cu flash	Cu	Cupro Nickel	>16, 250 psi	(1) VT
AD99	(b)	0.0005 Fe	Ni (c)	Fe plated Cb	6, 400 psi	(1) LKR
AD995	(b)	0.0005 Fe	Ni (c)	Fe plated Cb	4, 200 psi	(2) LKR
AD995 (pol)	(b)	0.0005 Fe	Ni (c)	Fe plated Cb	1, 200 psi	(2) LKR
AD995	(b)	0.0005 Cr	Ni (c)	Fe plated Cb	600 psi	(2) LKR

(a) VT indicates leak rate less than  $10^{-9}$  Torr 1/sec. LKR indicates leaker. Also see Appendix A.

(b) Evaporated thin films  $Ti < 1 \mu$ , then  $Mo > 1 \mu$ .

(c) Coast Metals Alloy 52 brazed at 1850°F, no hold time.

(>) Specimen broke in the ceramic.

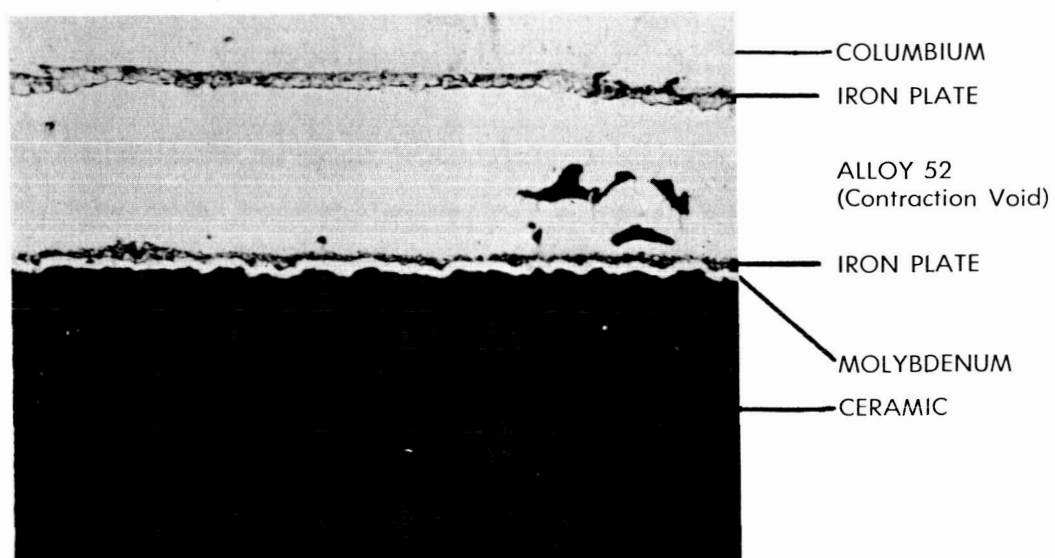
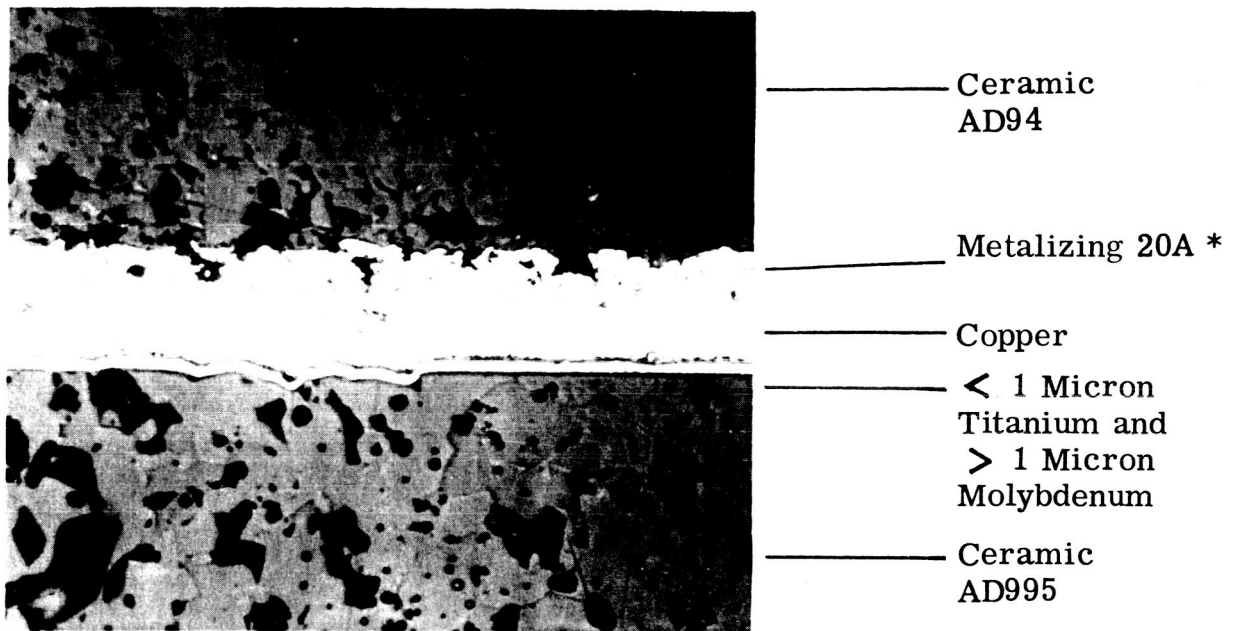


FIGURE V-10. Photomicrograph of Thin Film Metalizing  
With Alloy 52 Braze (X400)



\*Eitel - McCullough proprietary Metalizing

FIGURE V-11. Photomicrograph of Thin Film Metalizing With Copper Braze (X400)

## H. ELECTROFORMED SEALS.

No seals fabricated.

### CONCLUSIONS:

1. The facility development portion of this program is behind schedule. This is due to the upgrading which has been required to conform with the purity goals set by NASA and Westinghouse. These goals are now adequately defined and the facility as described in this report shows promise of meeting them. Completion of the facility by late April is forecast.
2. Most material specifications and ordering are complete. The ordering of the K and NaK in MSA "hot trap" containers will complete this item.
3. Preliminary work on seals has resulted in two promising metalizing compositions which remain vacuum tight with nickel alloy brazing (suitable for K and NaK). Thin film metalizing shows promise but associated brazing systems require further development.



References:

- LB20 Fox, C. W.; Gilliland, R. G., "Progress Report on Brazing of Columbium", ORNL 61-7-24, July 1961.
- LB24 Young, W. R., "Alloy Systems for Brazing of Columbium and Tungsten", ASD TR 62-592, Contract 33(616)7484, p. 81, January 1962.
- LB145 Stoddard, S. P.; Cowan, R. E., Ceramic Materials for Nuclear Thermionic Converters, Private Communication Los Alamos Laboratory, May 1963.

## SECTION VI

### TEST CONSIDERATIONS

#### A. FATIGUE.

Two photographs of the fatigue test arrangement are shown in Figures VI-1 and VI-2. The first figure shows the SF1U fatigue machine and 5-to-1 stress multiplying fixture and the second shows a set of specimen grips with a special strain gaged specimen in place. Specimen eccentricity, with respect to the center line of the grips, is held to less than 0.00025 inch. A specimen, previously calibrated in a tensile machine, is used with a visicorder and amplifier (shown in Figure VI-1 foreground) to check the dynamic calibration of the fatigue machine. The instrument in the foreground of the SF1U machine is used to measure dynamic creep, and the instrument on the right is used to control and record specimen temperature.

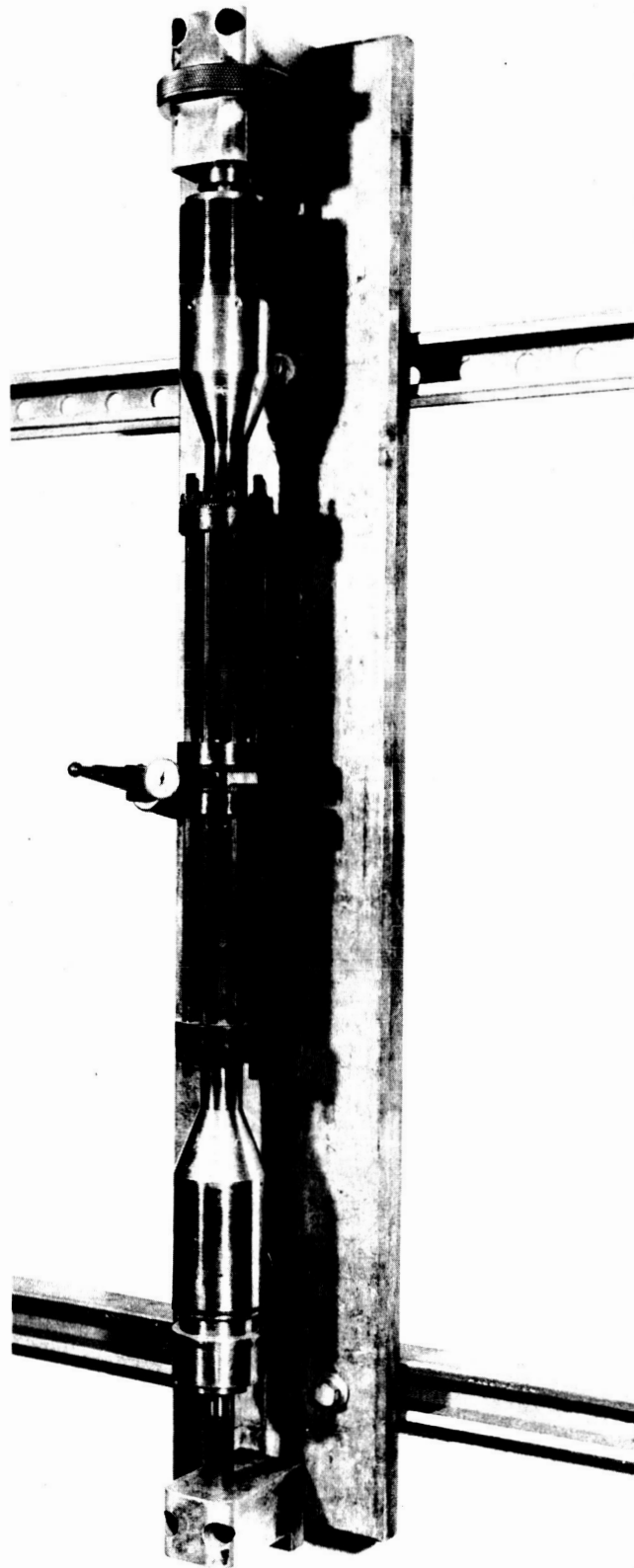


FIGURE VI-1. Fatigue Test Specimen Grips With Special Strain Gaged Specimen Positioned For Test

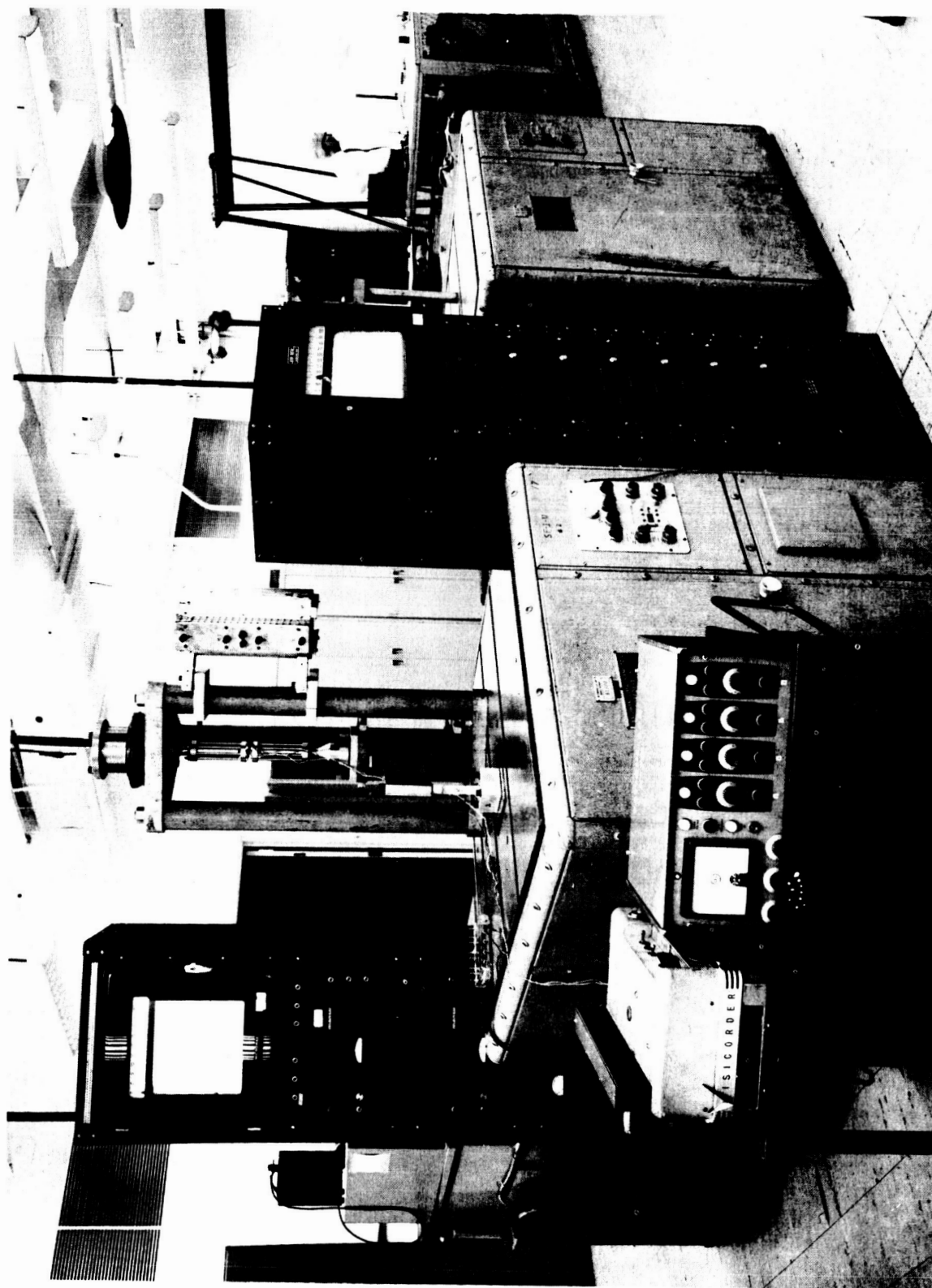


FIGURE VI-2. Type SF1U Fatigue Machine and Auxiliary Instrumentation and Test Equipment

## B. VACUUM CREEP CHAMBER LEAK RATES.

The major gas sources within high temperature vacuum creep test chambers are:

- 1) Leakage
- 2) Products of thermally degraded oil
- 3) Sorbed gases on surfaces
- 4) Chemical reaction products
- 5) Products resulting from degassing of the hot specimen

Since all the gaseous products are available for reaction with the test specimen and thus influence the results of the test, it is important to identify the species present and the transfer rates associated with them during testing.

If an assumption is made of  $10^{-5}$  mm Hg as the maximum pressure, and an actual pumping speed of 50 liters/sec is available, a calculation indicates a maximum leakage rate of the order of  $5 \times 10^{-4}$  cc-atm/sec from the equation

$$Q = SP$$

where: Q is the quantity of gas transferred per second

S is the pumping speed, and

P is the pressure of the system.

With transfer rates of this order of magnitude, the identification of gas species transferred can be made and their associated rates measured through the use of a cryogenic gas collection pump, 4.2°K located at the exhaust of the diffusion pump. At the above gas transfer rates, no difficulty would be expected in making such measurements. Because of the pumping speed of the available cryogenic pump, the direct measurements of the transfer rates of the above magnitude can be made only by utilizing the cryogenic pump following the diffusion pump where the gas transferred exists at a higher pressure and thus can be removed at a rate comparable to that associated with its removal from the creep test chamber by the diffusion pump. In order to perform a similar test by cryogenically pumping the creep test equipment directly, a cryogenic pump with a pumping speed of the same magnitude as the oil diffusion pump would be required. A suitable cryogenic pump to do this would represent a specialized pump and was beyond the scope of the leak rate investigation.

After prolonged pumping, leak rates for each vacuum creep chamber to be used for creep testing were measured with the cryogenic pump. Typical room temperature leak rates observed were  $2$  to  $5 \times 10^{-5}$  cc-atm/sec; at  $1600^{\circ}\text{F}$ , these values increased to approximately  $1 \times 10^{-4}$  cc-atm/sec. This increase at the high temperature was associated with increases in  $\text{H}_2$ ,  $\text{CO}$ , and hydrocarbon gases.

A typical analysis of gases transferred is shown in Table VI-1.

If the diffusion pump oil is a source or sink for gas, it would be reasonable to assume that the gas is in solution and is near equilibrium conditions. The worst condition is found when the gas dissolved in the diffusion pump oil is in equilibrium with the pressure on the mechanical pump side of the diffusion pump which is normally maintained in the range of  $10$  -  $20$  microns. Typical solubility values for  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{N}_2$  and  $\text{O}_2$  in the diffusion pump oil are about  $10\%$  volume of gas at one atmosphere of pressure per volume of oil. Assuming the quantity of gas in solution would decrease linearly with pressure (Henry's law), the quantity of soluble gas at  $10$  microns pressure would be approximately  $10^{-6}$  cc-atm of gas per cc. of oil. Since the pump contains approximately  $10^3$  cc of oil, the maximum gas content of the above species dissolved is of the order of  $10^{-3}$  cc-atm. Total quantities of gas collected over  $1200$  sec was of the order of  $10^{-1}$  cc-atm. Therefore, the oil in the diffusion pump could not retain more than about  $1\%$  of the above species. Carbon dioxide is about ten times as soluble as nitrogen and thus only  $10\%$  of the carbon dioxide collected could have been dissolved in the oil. The heavy hydrocarbon gases are approximately  $100$  times as soluble as nitrogen and thus a more rigorous study would be required to definitely establish their source.

The previous discussion suggests that a cryogenic pump can be used successfully to measure gas rates by collecting the exhaust to the oil diffusion pump. However, if there are pumping mechanisms associated with the vacuum test chamber, the measured rates will be low by the fractional ratio of the pumping speed associated with the diffusion pump to the total pumping speed of both the diffusion pump plus pumping mechanisms within the creep test equipment.

Ionization gages were attached to one test chamber in order to measure the gas rate by pressure increase in the vacuum chambers. The pressure sensing instruments used in these vacuum creep systems are Pirani gages which are not as sensitive as ionization gages. The ionization gages showed the ultimate pressure to be of the order of  $1 \times 10^{-7}$  mm-Hg in the chambers at room temperature while pumping. The chamber pressures increased to approximately  $1.5 \times 10^{-7}$  mm-Hg when the temperature was raised to  $1600^{\circ}\text{F}$ . Leak rates at both temperatures were measured by timing the rate of rise from  $1 \times 10^{-6}$  to  $5 \times 10^{-6}$  mm-Hg with the system valves disconnecting the